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Assessing ASR Potential of Hardened Concrete and The Use of Rice Husk Ash to Mitigate ASR

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ASSESSING ASR POTENTIAL OF HARDENED CONCRETE AND THE USE OF RICE HUSK ASH TO MITIGATE ASR

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ABSTRACT

This two part research study was created to first develop a new process using existing test methods to determine the Alkali-Silica Reaction (ASR) potential of hardened concrete that is exposed to external alkalis and secondly to determine the ASR mitigation potential of using rice husk ash (RHA) as a supplementary cementitious material (SCM). The first part of the research study was completed by using a new aggregate extraction procedure that uses a combination of hydrochloric acid treatments and abrasion to remove the cement paste from the aggregate. This extracted aggregate was then crushed down to the specified sizes and used in an ASTM C 1260 test. The calibration of the test was done by extracting three different types of aggregate from ASTM C 1293 samples from a previous study at Clemson University that had ASR problems and then using the extraction process to remove enough aggregate to conduct a post removal ASTM C 1260 test. The results of the post removal ASTM C 1260 test were then compared to the initial ASTM C 1260 tests that were run on the three control aggregates. The new process was successful in the sense that the aggregates were classified with the same reactivity for the pre and post removal ASTM C 1260 tests. This process was then used on cores from two airports; both of these airports are known to have ASR problems. The test was again successful in determining the reactivity of the aggregates as potentially having a deleterious behavior. As for the determination of the mitigation potential of RHA used as an SCM, this was done by first physically characterizing the material, determining the pozzolanic activity index, and then using the RHA in an ASTM C 1567 test to determine the mitigation potential. The results showed that the material has a low specific gravity with a high

surface area and high silica content. The pozzolanic activity index was the highest at a replacement level between 10 and 15% by mass replacement. The results of the ASTM C 1567 tests showed that as the replacement level increases so does the expansions, which is a clear sign that the use of RHA failed as an ASR mitigator. The reason for this is unknown, but may be due to either the vesicular structure of the RHA grains creating direct routes or pathways for the external alkalis to be transported or the chemical make-up of the ash causing an interaction with the reactive silica and external alkali

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INTRODUCTION

This research thesis is comprised of two parallel research studies that relate to different aspects of ASR. The first section will deal with the ASR testing of hardened Portland cement concrete and the second section will deal with the physical characterization and the ASR mitigation potential of the use of RHA from a new process as a secondary cementitious material. Each section will have its own set of conclusions, but at the end of the paper there will be ultimate conclusions drawn from both of the studies.

Alkali-silica reaction (ASR) has been studied since the 1940's, starting with the research done by Thomas Stanton (22). Stanton's research found that failures in Portland cement concrete due to excessive expansions were caused by a chemical reaction between the alkalis in the cement paste and certain reactive forms of silica located within the aggregate. There is a significant amount of knowledge developed through research available on the mechanism of ASR, but relating the tests done on standard mixtures to the actual hardened concrete may not be possible due to the fact that field conditions of the hardened concrete may not be the exactly same as the standard laboratory tests. The reason for this study is to determine a process using existing test methods to determine the ASR potential of hardened concrete. To achieve this, cores were taken from the existing concrete affected with ASR. The cores were crushed down to extract the aggregate by the use of a combination of abrasion and hydrochloric acid treatment to dissolve the mortar matrix. The extracted aggregate is then crushed to finer sizes and

used in a standard and a modified ASTM C 1260 test to determine the existing reactivity of the aggregate.

To prevent this measure from being necessary there have been many proposed mitigation measures for ASR such as the use of low-alkali cements, the use of secondary cementitious materials (SCM), and the use of lithium admixtures. One type of natural SCM that has limited knowledge on its potential mitigation is rice husk ash (RHA). Another focus of this study was to determine the mitigation potential of ASR with the use of RHA as a cement replacement as well as the mechanical and chemical properties of RHA. There are two reasons to use RHA: 1.) compared to the other SCMs there is much less known about RHA and its effectiveness in reducing expansions caused by ASR and 2.) the RHA used in this investigation has a significantly lower carbon content compared to other sources previously evaluated. The controlled burning of rice husks produces RHA. This use of RHA in concrete is an environmentally friendly alternative to open heap burning of the husks, which produces harmful gases and has become an illegal practice in the United States. The estimated current world production of the rice grain itself is about 600 million tones, with $1/5^{th}$ of the 600 million tones being the husk itself and when it is burned the ash produced is about $1/5^{th}$ of that amount. RHA has been experimented with as an SCM because of its high silica content, which in turn helps to increase the pozzolanic reaction. It has been postulated by Hasparyk that "the presence of silica fume and RHA reduced the cracking that commonly develops in mortars with reactive aggregates", but do to the fact that the RHA used in this study is of a new

process this can not be assumed (9). It has also been seen that the "physicochemical properties of RHA are similar to that of silica fume, although the former may be even more strongly pozzolanic than the latter" (9). This indicates that RHA should be a good mitigator of ASR induced expansions, due to the fact that silica fume has been known to be successful at doing so. To determine the potential for mitigation of ASR expansions and cracking the same modified ASTM C 1260 test was run, which will be discussed more in the materials and methods chapters of both of the sections.

SECTION 1 (ASR TESTING OF HARDENED CONCRETE)

1. INTRODUCTION

1.1 PURPOSE

The purpose of developing a new process using existing test methods to determine the ASR potential of hardened concrete is that there are currently no established tests to assess this potential. There are tests to determine the reactivity of an aggregate, but this does not take into account the alkalis that are and could be added to the concrete system in the field such as from external ground sources, and runway/roadway deicers. With these added variables, a new test method or process using existing methods is needed to determine the ASR potential of existing concrete in the field.

1.2 BACKGROUND

Since the 1940's ASR has been studied starting with the research done by Thomas Stanton. It was found that excessive expansions can be caused by a chemical reaction between the alkalis in the cement paste and certain reactive forms of silica located within the aggregate. Test methods such as ASTM C 227 have been around since the 1950's; however this test was designed to determine alkali reactivity between cement-aggregate combinations, but was later determined to not accurately identify all reactive aggregates. This led to the development of other tests such as ASTM C 1260, ASTM C 1293, and ASTM C 1567, which are designed to determine reactivity of aggregates or mitigation potential of ASR through the use of secondary cementitious materials. These tests have their individual merits. A potential problem with these tests is that if an aggregate is classified as non-reactive and used in the field map-cracking associated with ASR appears after a few years. This problem may not be identified by these tests, because

these tests do no take into account the field conditions, which may increase the alkali content that was not included in the laboratory tests. This is because alkalis can penetrate the system externally through either adjacent ground sources or the application of runway/roadway deicers. Currently, there are no tests that can determine the potential of ASR in hardened concrete that is subjected to this external supply of alkalis. This is the reason behind the need for a new process using existing test methods to determine the ASR potential of concrete with an external supply of alkalis.

1.3 OBJECTIVES

The objectives of this research were to:

- Develop a process to remove aggregate from cores taken from hardened concrete.
- Determine the reactivity of the extracted aggregate taken from the hardened concrete using standard and modified ASTM C 1260.

1.4 SCOPE OF RESEARCH

The scope of the research includes the calibration of the process by first conducting ASTM C 1293 and C 1260 tests on the aggregate and then removing the aggregate through the created procedure from the ASTM C 1293 prisms, which are then used in an ASTM C 1260 procedure to determine the current reactivity of the aggregate. The pre-removal ASTM C 1260 test results were then compared to the post-removal ASTM C 1260 test results. Sections of the post-removal ASTM C 1260 bars were then epoxy impregnated for SEM microscopy with EDX analysis. Once the test was calibrated,

cores taken from two airport taxiways, which are both known to have ASR problems, were then tested using this new procedure of removing the aggregate and using ASTM C 1260 to test the aggregates current reactivity.

2. LITERATURE REVIEW

2.1 ASR IN HARDENED PORTLAND CEMENT CONCRETE

2.1.1 Introduction

ASR or alkali-silica reaction has been studied since the 1940's, starting with the research done by Thomas Stanton, with his publication for the American Society of Civil Engineers published in 1940 (22). Stanton's research found that failures in Portland cement concrete due to excessive expansions were caused by a chemical reaction between the alkalis in the cement paste and certain reactive forms of silica located within the aggregate. More recently it has been stated that

> "Alkali-silica reaction (ASR) is a chemical reaction between the hydroxyl ions in the pore water within concrete and certain forms of silica that occasionally occur as part of aggregate resulting in the formation of alkali-silica solutions. This type of reaction is known to cause abnormal expansion in concrete, and as a result, extensive cracking occurs" (17).

This cracking shows up in the form of map cracking or pattern cracking seen in Figure 2.1, this distress will eventually lead to the ultimate failure of the concrete system, but signs of the distress may not become visible for at least five to ten years after placement.

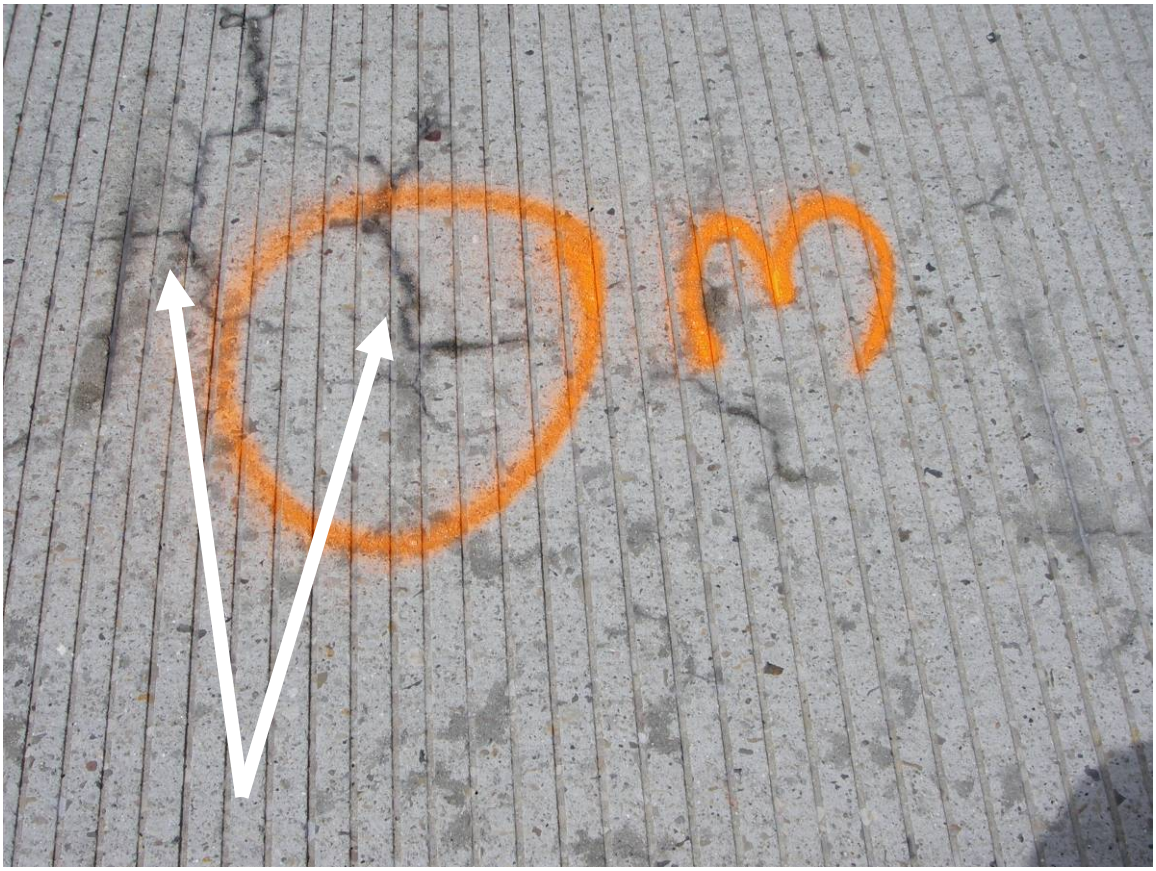

Figure 2.1: Map Cracking in Portland Cement Concrete taken from: Airport #1

It has been stated that "ASR distress takes a number of years to manifest in field conditions, depending on the reactivity of the aggregate and on exposure conditions" (19). Stanton's research also determined 5 main factors that influence the alkali-silica reaction and those were: 1.) nature of reactive silica, 2.) amount of reactive silica, 3.) particle size of reactive material, 4.) amount of available alkali, and 5.) amount of available moisture (22). For the reaction to first take place it has been found that "the ASR reaction needs

several components to take place: alkali (supplied by the cement, although external sources can exist), water (or a high moisture content), and a reactive aggregate" (13). For the aggregate to be reactive, it depends on the type of silica contained within the

2.1.2 Use of Secondary Cementitious Materials as an ASR Mitigator

The uses of pozzolans have been considered to reduce ASR expansions. Based on Chatterji's hypothesis that calcium hydroxide is a necessary factor in the cause of ASR he stated that "small addition of pozzolana delayed the onset of expansion whereas a larger addition of the same pozzolana completely suppressed the expansion" (5). This is due to the pozzolanic reaction in which the silica present in the SCM or pozzolana reacts with the excess calcium hydroxide to form C-S-H gel which generates increased strength. This reduction in calcium hydroxide helps to slow down or completely stop the excessive expansions. The alkali content of the SCM is a factor in its effectiveness in ASR mitigation as well as the recommended replacement level. In a report by Duchesne and Berube for the ACI Materials Journal they state that "SCM alkali content is critical because a higher content calls for higher cement replacement levels", but "excessive alkali content may make the SCM totally ineffective, even with relatively high SCM contents" (7). Recommended SCMs can include class F fly ash, Ground Granulated Blast Furnace Slag (GGBFS), silica fume (SF) and class N natural pozzolans such as metakaolin or RHA. Certain chemical limitations and potential problems have been suggested in a review of literature entitled *Alkali-Silica Reaction Mitigation: State of the*

Art and Recommendations present in the ACI Materials Journal. This review gives the following recommendations (10):

- Class F fly ash should have less than 10% CaO, less than 1.5% alkalis, and a maximum LOI of 6% with replacement levels of between 15 and 45 percent,
- GGBFS should have low alkali levels with replacement levels starting at 40% and increasing,
- Silica fume if not dispersed correctly can lump together in the fresh concrete and these lumps can eventually react with the alkalis in the same way reactive aggregates do, replacement levels are typically around 10%,
- Class N pozzolans or natural pozzolans should have less than 2% CaO, almost no alkalis, and a LOI less than 4%, with replacement levels between 10 and 25 percent.

Compared to the other SCMs there is much less known about RHA and its effectiveness in reducing expansions caused by ASR. It has been seen that the "physicochemical properties of RHA are similar to that of silica fume, although the former may be even more strongly pozzolanic than the other" this indicates that RHA should be a good mitigator of ASR induced expansions, due to the fact that silica fume has been known to be successful at doing so (9). In Hasparyk's study he found that "the presence of SF and RHA reduced the cracking that commonly develops in mortars with reactive aggregates, highlighting the strong relationship between gel composition and expansion" and he further goes on to state that "these pozzolans also significantly changed the gel composition" (9). The changing of compositions of the gels causes the gel to be non-

expansive or less expansive and due to this there are reductions in ASR induced expansions and cracking.

2.1.3 Use of Lithium Based Compounds to Mitigate ASR

Along with the use of SCMs and low-alkali cement, penetrating lithium nitrate compounds are also used to mitigate the excessive expansions of ASR. Lithium nitrate compounds are either added to the concrete during mixing or they can also be added to the hardened concrete by spraying the compound on the hardened concrete and allowing the lithium nitrate to penetrate the concrete. The penetration of the lithium nitrate may not always be sufficient to mitigate the ASR induced expansions, so research is being done to determine the best methods of insuring sufficient penetration. One such method is by driving lithium ions into the hardened Portland cement concrete using electrical fields.

2.1.4 Conclusion

ASR is a function of three main elements: alkali, water, and reactive silica. As it has been found that for it to first take place "the ASR reaction needs several components to take place: alkali (supplied by the cement, although external sources can exist), water (or a high moisture content), and a reactive aggregate" (13). Stanton's research also determined 5 main factors that influence the alkali-silica reaction and those were: 1.) nature of reactive silica, 2.) amount of reactive silica, 3.) particle size of reactive material, 4.) amount of available alkali, and 5.) amount of available moisture (22). The use of

SCMs and lithium compounds has been found to be successful in mitigating ASR expansions, but ASR is still a major problem both mechanically and financially in today's society. Even though ASR has been researched thoroughly there is still no one immediate fix all solution for this deleterious mechanism.

The mechanism of ASR has been well studied as previously seen, but a limitation to this research has been the studying of this reaction using controlled environments. The increasing usage of potassium acetate deicers has created this need to determine the ASR potential of hardened concrete, because these deicers provide an external source of alkalis that was not compensated for in the initial research. The reason for the creation of a new process using existing test methods to determine the ASR potential of hardened concrete is due to this fact.

3. MATERIALS AND METHODS

3.1 INTRODUCTION

The materials used in this new test method were cores or samples of existing concrete. The cores, four inch diameter, in this study were taken from two airport taxiways. As for the existing samples they were taken from another study conducted at Clemson University (20-22). These samples were taken from an ASTM C 1293 test, so the dimensions for the samples were 3"x 3"x12". These samples, being the cores and the ASTM 1293 prisms, were then broken down and the aggregate removed. The process for removing the aggregate will be discussed in the following section. This removed

aggregate was then pulverized into the correct aggregate gradation for an ASTM C 1260 test, and then the test was run to determine the current reactivity of the removed aggregate.

3.2 AGGREGATE REMOVAL FROM HARDENED PORTLAND CEMENT CONCRETE

The process for removing the aggregate from the hardened Portland cement concrete involved the use of 1N hydrochloric acid to breakdown the mortar matrix. The samples, either being the cores or the ASTM 1293 prisms, started off by being cut into thinner sections with a concrete saw so that the samples could then be mechanically broken apart using a 3lb hammer. The smaller chunks of the concrete were then placed in a 1N hydrochloric acid bath for between 1 and 2 weeks. The samples were then taken out washed over a #4 sieve and then placed in a Micro-Deval device with 245 gms of balls per container and then run for 2 hours. The samples were then taken out and washed again over the #4 sieve and then the clean aggregate was removed if any were present. The determination of "clean" aggregate, that is aggregate that has small or no quantities of cement paste, was done by the researcher, with the limit of Portland cement paste on the aggregate covering less than 25% of the total aggregate surface area. The remaining aggregate-mortar chunks were then placed back in the container along with a liter of the 1N hydrochloric acid. The containers with the acid and aggregate-mortar chunks were stored in a constant temperature room with the temperature being 38°C. This process of cleaning the aggregate, running it through the Micro-Deval, and either clean aggregate removed or "dirty" aggregate being placed back in the hydrochloric acid solution

continued until enough clean aggregate was removed to run a standard and modified ASTM C 1260 test. The length of the process is dependent on the concrete being tested. Samples have been seen to take as little as 3 months and can take up to 1 year and on to extract the aggregate. An example of the visual progression of the removal process is displayed in Figure 3.1 for the New Mexico sample that was removed from previously made ASTM C 1293 prism that was soaked in the 1N sodium hydroxide.

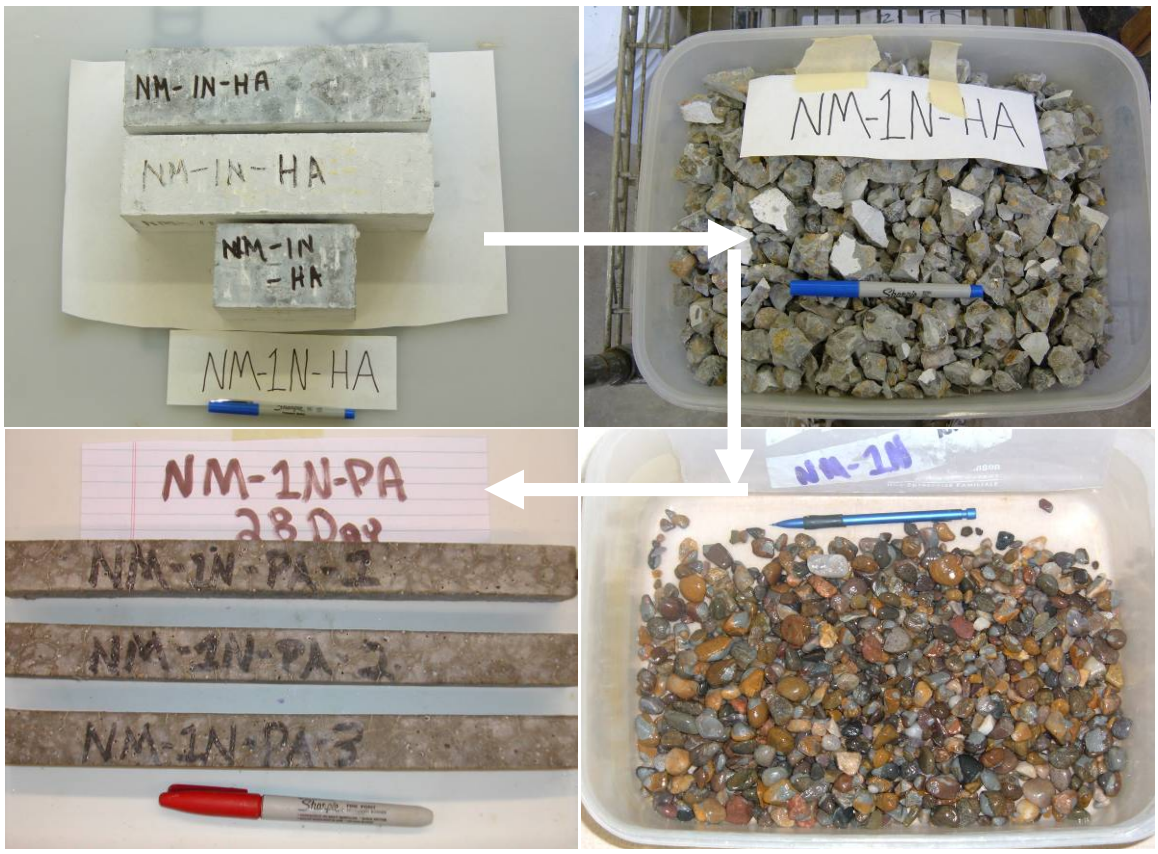

Figure 3.1: Example of the Visual Progression of Aggregate Removal

For all of the other removed aggregate visual progression images look in the appendix section B.

3.3 TEST MATERIALS

3.3.1 Cement

The Portland cement used in this study was high-alkali Type I Portland cement with the chemical composition listed in Table 3.1.

Table 3.1: High-alkali Type I Chemical Composition

Oxide	**Percent (%)**
SiO_2	19.74
Al_2O_3	4.98
Fe_2O_3	3.13
CaO	61.84
MgO	2.54
SO_3	4.15
LOI	1.90
$Na_2O_{equivalent}$	0.82
K_2O	0.84
Na_2O	0.27
Insoluble Residue	0.25
C_3A	8.97
C_3S	46.6
Autoclave Expansion	0.12

3.3.2 Aggregate

There were three different types of known aggregate removed from the ASTM 1293 samples and two types of unknown aggregate removed from cores taken from two airports. The known aggregates used were a quartzite from South Dakota, an argillite from North Carolina, and a rhyolite from New Mexico, all of these aggregates have a history of being reactive and the two airports have known ASR problems. Some of the

aggregates properties are listed in Table 3.2. A portion of the aggregates removed were examined using a scanning electron microscope (SEM) with an energy-dispersive X-ray spectroscopy (EDX) attachment to determine if any remaining reaction products were still left within the aggregates. The aggregate samples were prepared by vacuum penetrating with epoxy and once the epoxy had set the samples were polished using multiple grit pads. It was determined through this investigation that no remaining reaction products remained within the aggregate. An example of the visual display of this data is shown in Figure 3.2, with the rest of the aggregate investigations being found in the appendix section. The aggregates removed from the two airports were known to have ASR problems due to visual inspection and other previous investigations. The visual inspection of airport #1 showed map cracking in the main runways, this is shown in Figure 2.1 and Figure 3.3.

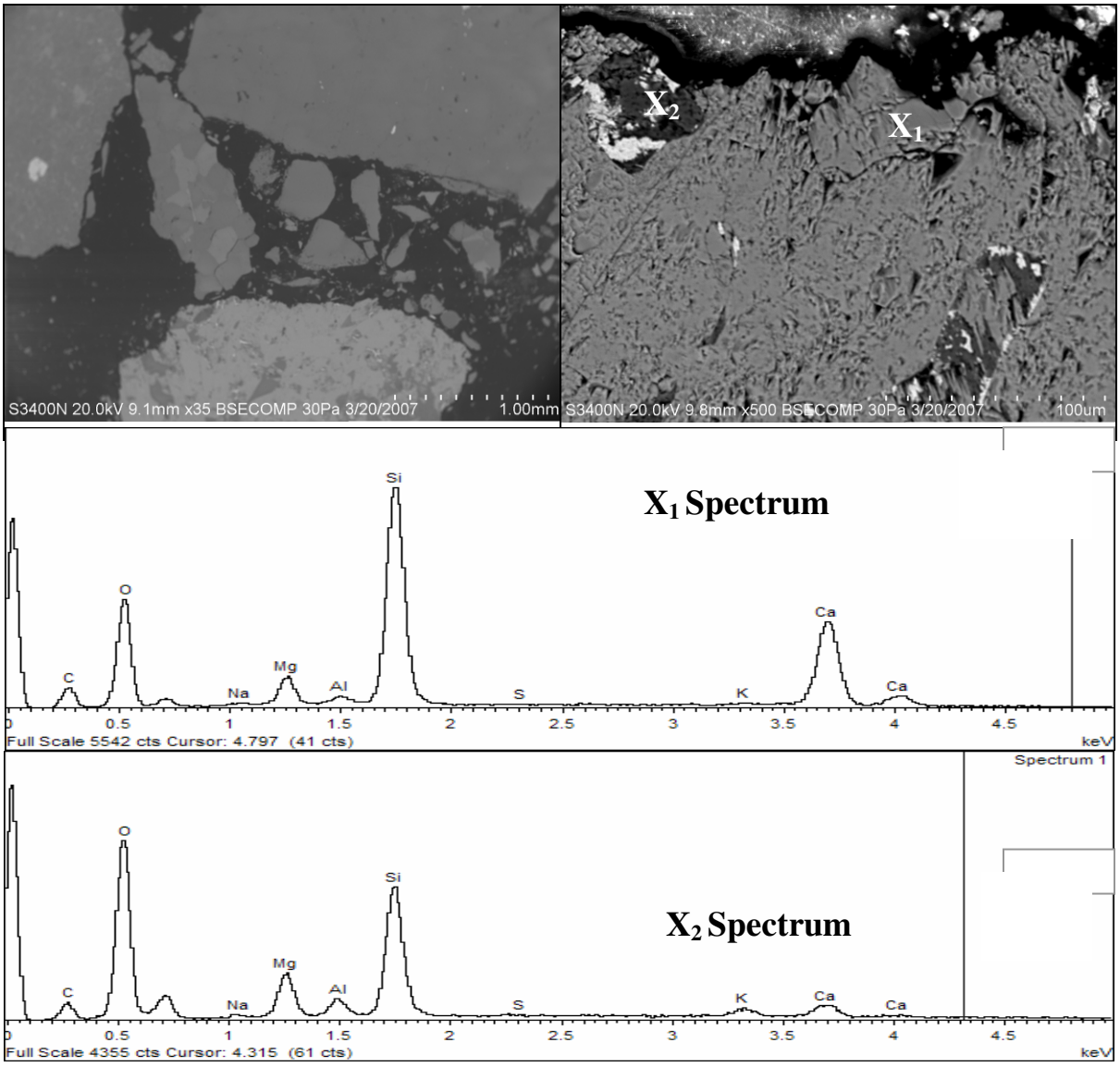

Figure 3.2: SEM and EDX Investigation of the Removed New Mexico Aggregate Alone

Table 3.2: Aggregate Physical Properties

Aggregate Property	SD, Quartzite	NC, Argillite	NM, Rhyolite
Water Absorption, %	0.42	0.344	1.087
Bulk Specific Gravity (OD)	2.51	2.75	2.60
Bulk Specific Gravity (SSD)	2.52	2.76	2.63
Dry Rodded Unit Weight, kg/m^3	1557.6	1566	1585.3

Figure 3.3: Map Cracking on the Airport #1 Taxiways

A previous investigation of cores taken from the 2nd airport showed that an ASR problem was also evident. This was due to a visual inspection that showed abundant map cracking and also a SEM with an EDX attachment analysis was done, which proved that ettringite was forming and also cracking through the aggregate was occurring, with both of these being obvious signs of an ASR problem. This can all be seen in Figure 3.4.

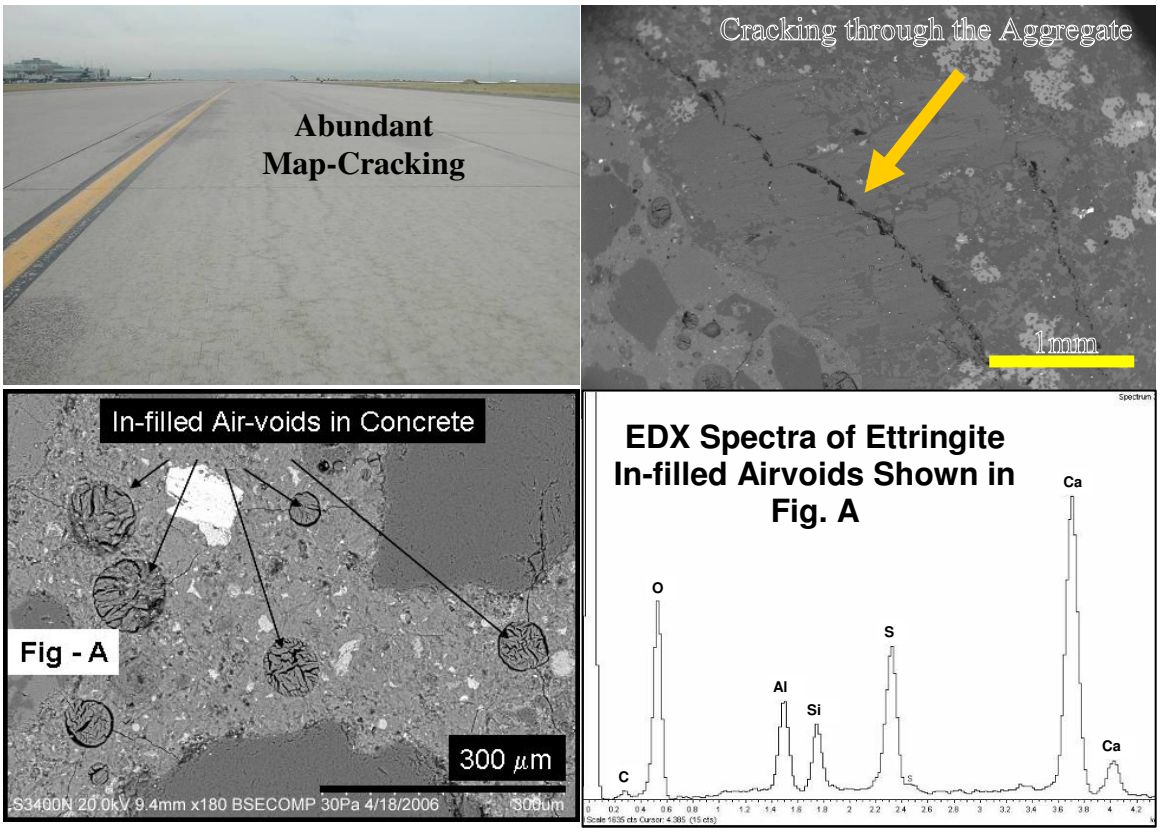

Figure 3.4: Initial Investigation of Airport #2 Taxiways

3.4 TEST METHODS

3.4.1 Standard and Modified ASTM C 1293 Test (Pre-Removal)

The standard and modified ASTM C 1293 test or the *Standard Test Method for Determination of Length Change of Concrete Due to Alkali-Silica Reaction* was conducted by a former Clemson University graduate student (20-21). This test was done according to the standard, which specifies storing the specimens vertically in a container with a small water reservoir that is not in contact with the prisms at 38℃. The modified part of the test involved storing prisms in solutions of 1N NaOH and potassium acetate, similar to the ASTM C 1260 procedure. The use of 1N NaOH was to ensure that the

Na_2O_e in the bars was still equivalent to 1.25% at all times during the test, due to the fact that some of the alkalis will leach out reducing the Na_2O_e in the standard ASTM C 1293 test. The modification using potassium acetate solution was done to determine the added effect of using a deicer, which is an external supply of alkalis, on the reactivity of the aggregates tested. "Pre-Removal" indicates that these samples were the ones that the aggregates were removed from. These samples, having a cross section of 3"x3", were made with the respected aggregate and left to cure overnight in a 100% relative humidity room. The next day the samples were removed and initial comparator readings were taken and then the prisms were placed in their respected solution (1N NaOH or potassium acetate). These solutions were then placed in a 38°C room and readings were taken according to the schedule in ASTM C 1293. All of these readings were converted into percentage increases and then compared to the limit of 0.04% at one year, which indicates potentially deleterious reactive aggregate above this limit.

3.4.2 Standard and Modified ASTM C 1260 Test (Pre-Removal)

ASTM C 1260 test or the mortar-bar method was conducted by a former graduate student of Clemson University on the three types of aggregate discussed previously (20-21). This test was done in the standard form along with a modified version, with the modification being the replacement of the 1N NaOH bath with a potassium acetate bath. "Pre-Removal" indicates that the test was done before the removal of aggregate from the ASTM C 1293 prisms. The test results give a baseline reactivity of the aggregates that is used to compare to the results from the ASTM C 1260 test (Post-Removal). The

comparison helps to determine if the aggregates lose reactivity over time while in hardened Portland cement concrete, or if the reactivity stays constant over time, and also this comparison is a calibration of the new procedure to determine the in-place potential of ASR in hardened Portland cement concrete. These mortar bars were made according to the ASTM procedure and left to cure overnight at 100% R.H. The day after the bars were made they were demolded and a length measurement was taken, then they were placed in a water bath overnight in an 80°C oven. The following day another reading was taken, which is used as an initial reading, and then the bars were placed in their respected solution (1N NaOH or potassium acetate). Measurements were taken a 3, 7, 10, 14, 21, and 28 days.

3.4.3 Standard and Modified ASTM C 1260 Test (Post Removal)

A second ASTM C 1260 test was conducted using the removed aggregate from the ASTM C 1293 prisms for the calibration (a visual description of this is in Figure 3.1) of the new procedure along with removed aggregates from cores taken from the two airports, which are known to have ASR problems. This test was also done by the standard along with a modification in a parallel test with the modification being the replacement of the 1N NaOH solution with a Potassium Acetate solution. These test results were then compared to the original ASTM C 1260 tests for the three types of aggregates, and for the other two aggregates the results were compared to field observations. These mortar bars were made according to the ASTM procedure and left to cure overnight at 100% R.H. The day after the bars were made they were demolded and a length measurement was

taken, then they were placed in a water bath overnight in an 80°C oven. The following day another reading was taken, which is used as an initial reading, and then the bars were placed in their respected solution (1N NaOH or potassium acetate). Measurements were taken a 3, 7, 10, 14, 21, and 28 days. The designations of the samples are shown in Figure 3.5 where X represents the type of aggregate used. For example, the mortar bars placed in the potassium acetate solution made from New Mexico aggregate that was removed from the prisms that were placed in potassium acetate have the name NM-PA-PA.

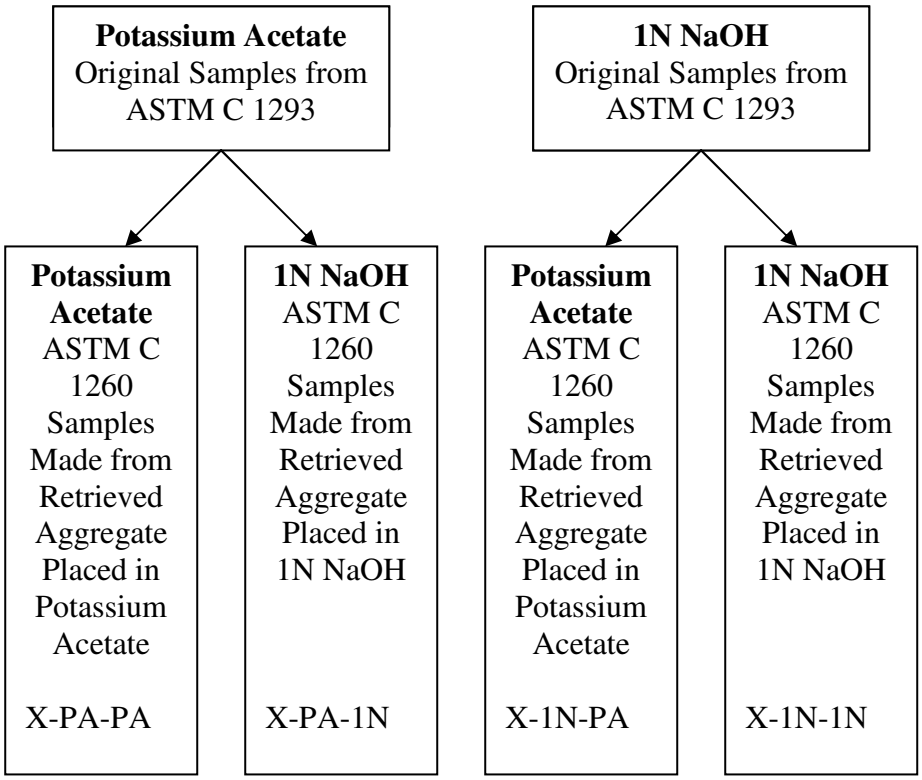

Figure 3.5: Visual Description of Removal and Retesting Process Along with Naming Examples

3.4.4 SEM Investigation (Post Removal)

SEM analysis was conducted at Clemson University's Electron Microscope Facility. The microscope used was a variable pressure Hitachi S-3400 SEM. Samples looked at were of the removed aggregate and also mortar bar sections taken from the Post Removal ASTM C 1260 samples. Samples were prepared by vacuum penetrating a two-part epoxy into either the pulverized aggregate samples or the sectioned mortar bar samples until visually no air bubbles were coming to the surface of the samples. The vacuum penetrating was done using desiccators and a vacuum pump. The samples were then placed in an oven until the samples hardened. Once the samples were hardened they were removed from the molds and then the samples were then sliced to expose the aggregate or mortar bar section surface. The samples were then polished to remove surface defects. A series of polishing pads of different grits were used to polish these samples on a polishing wheel. These samples were then ready to be analyzed using the SEM.

4. RESULTS AND DISCUSSION

4.1 STANDARD AND MODIFIED ASTM C 1293 TEST (PRE-REMOVAL)

The results from the ASTM C 1293 test along with the ASTM C 1260 test give a baseline reading of reactivity for the three different types of aggregate. It was determined through the ASTM C 1293 test that all three of the aggregates are classified as potentially deleteriously reactive. From the test, potentially deleteriously reactive indicates that the samples expanded on average more 0.04% at one year. From this, it was determined that all three of the aggregates are reactive, therefore, for this new test method to be

successful the results of the ASTM C 1260 test conducted using the removed aggregate should indicate the same reactivity. The results of the standard and modified ASTM C 1293 tests can be seen in Figures 4.1-4.6 for each of the aggregates.

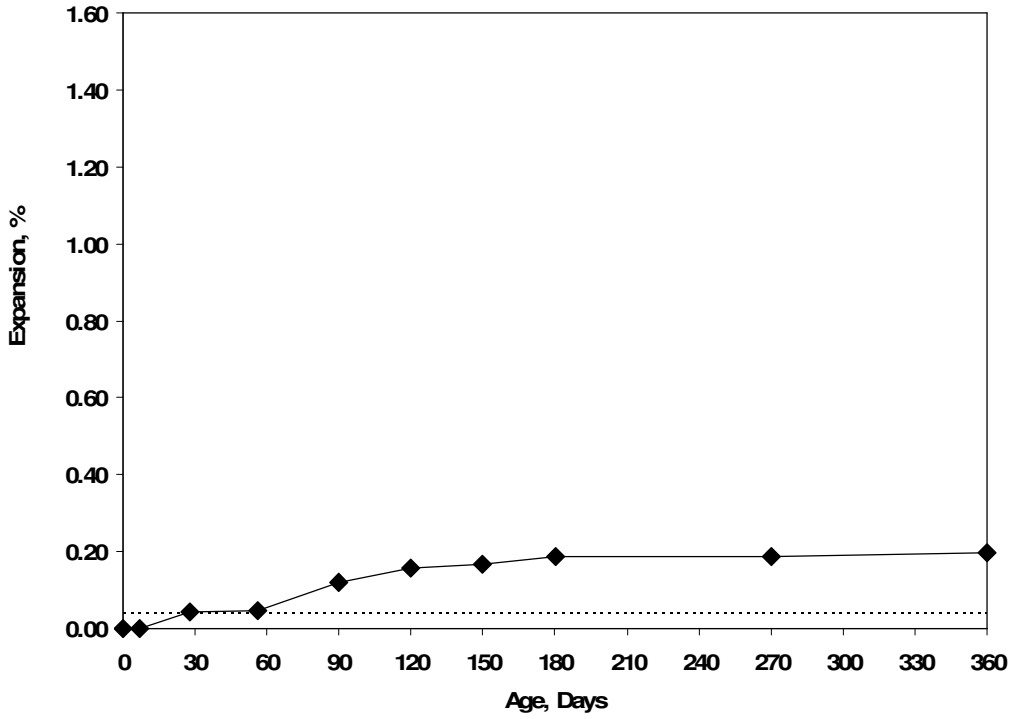

Figure 4.1: Standard ASTM C 1293 Test for the New Mexico Aggregate

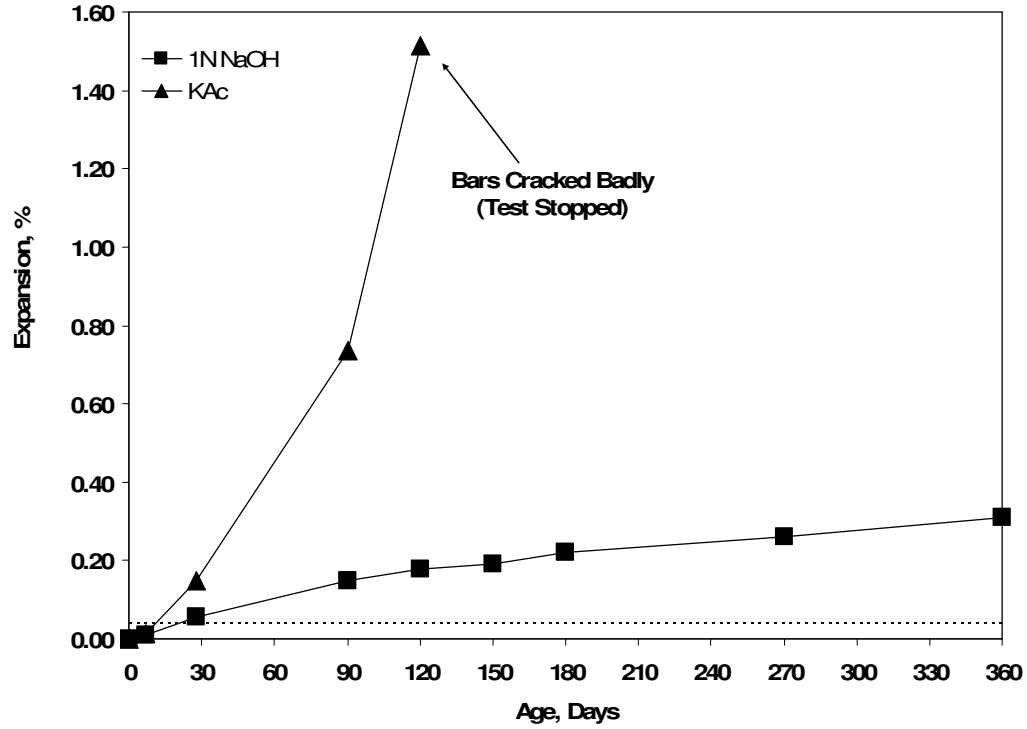

Figure 4.2: Modified ASTM C 1293 Test for the New Mexico Aggregate

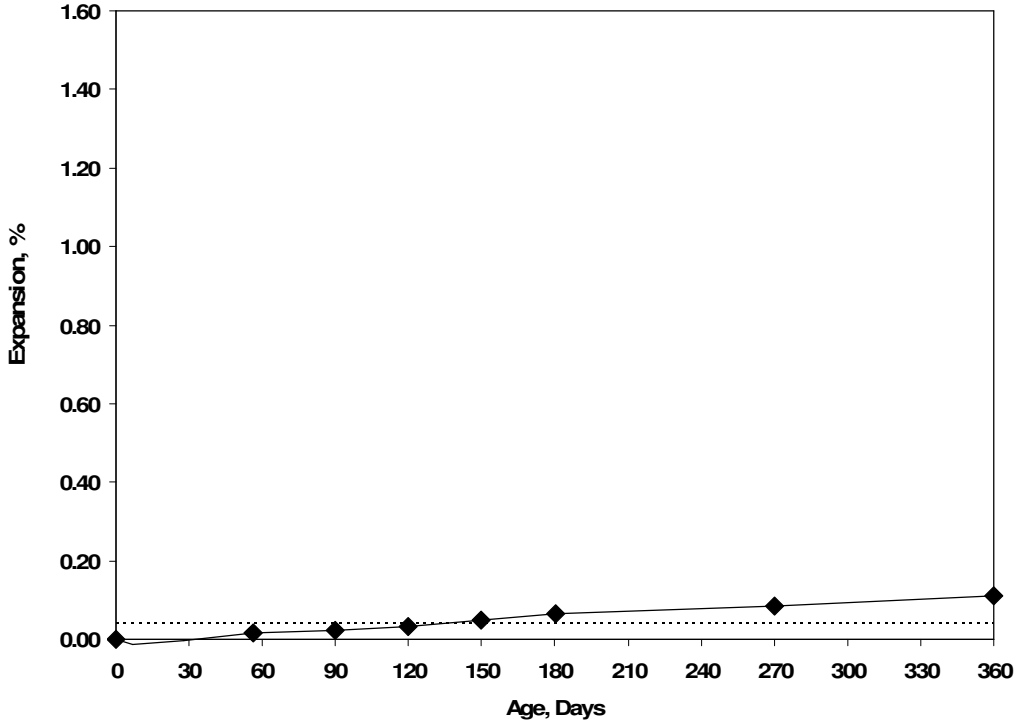

Figure 4.3: Standard ASTM C 1293 Test for the South Dakota Aggregate

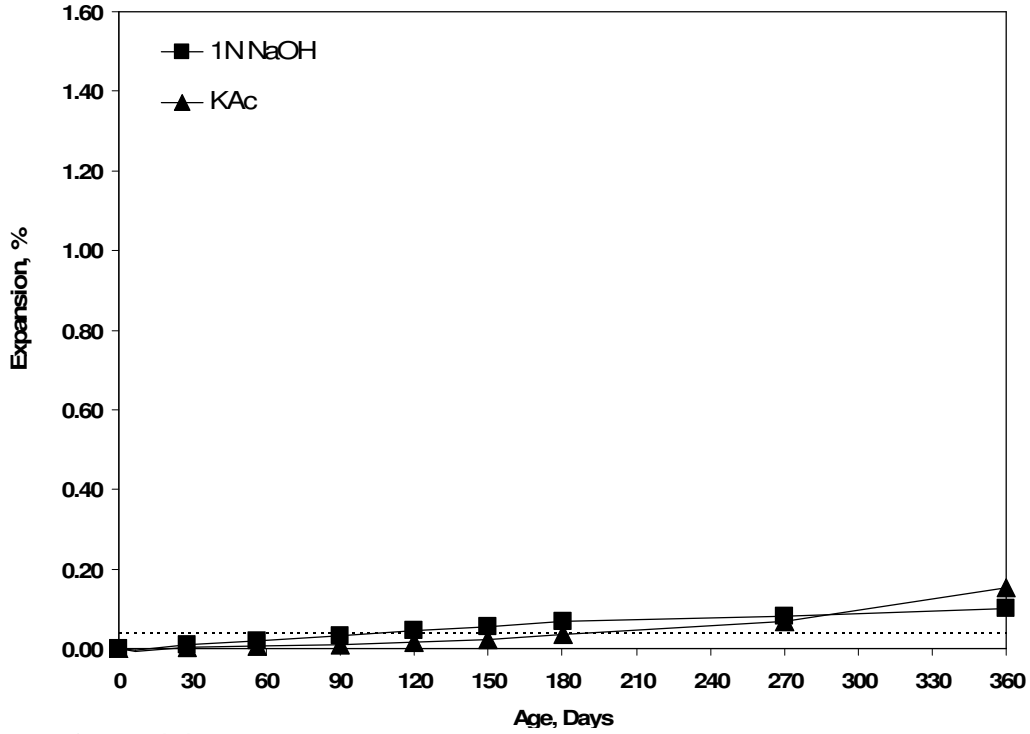

Figure 4.4: Modified ASTM C 1293 Test for the South Dakota Aggregate

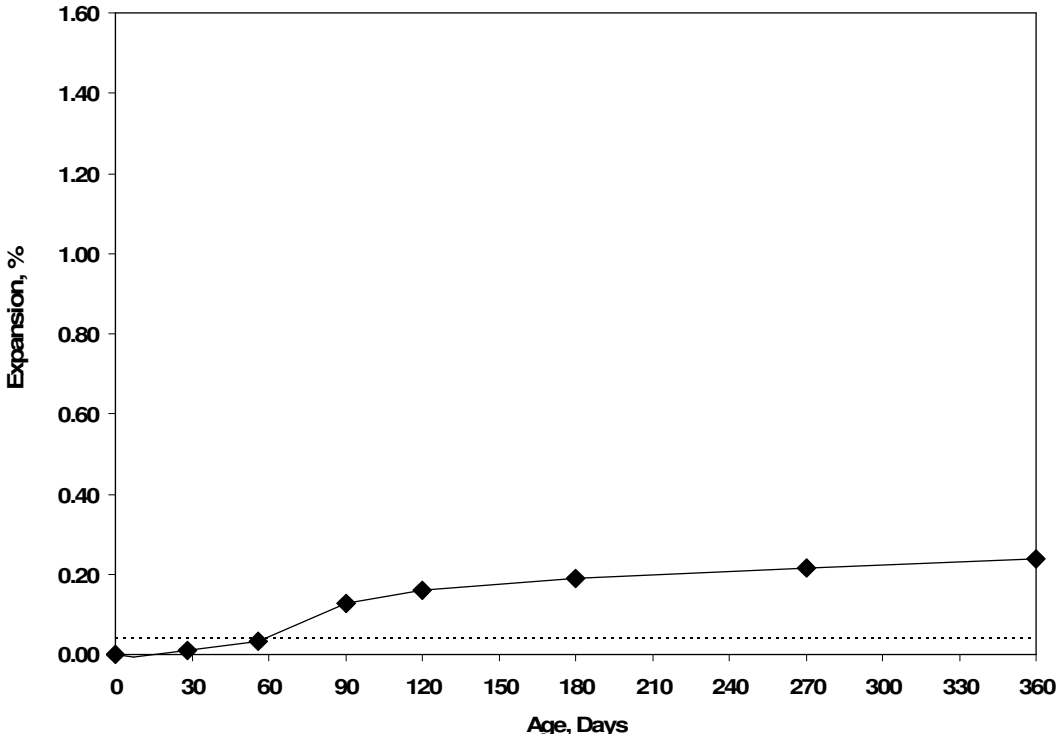

Figure 4.5: Standard ASTM C 1293 Test for the North Carolina Aggregate

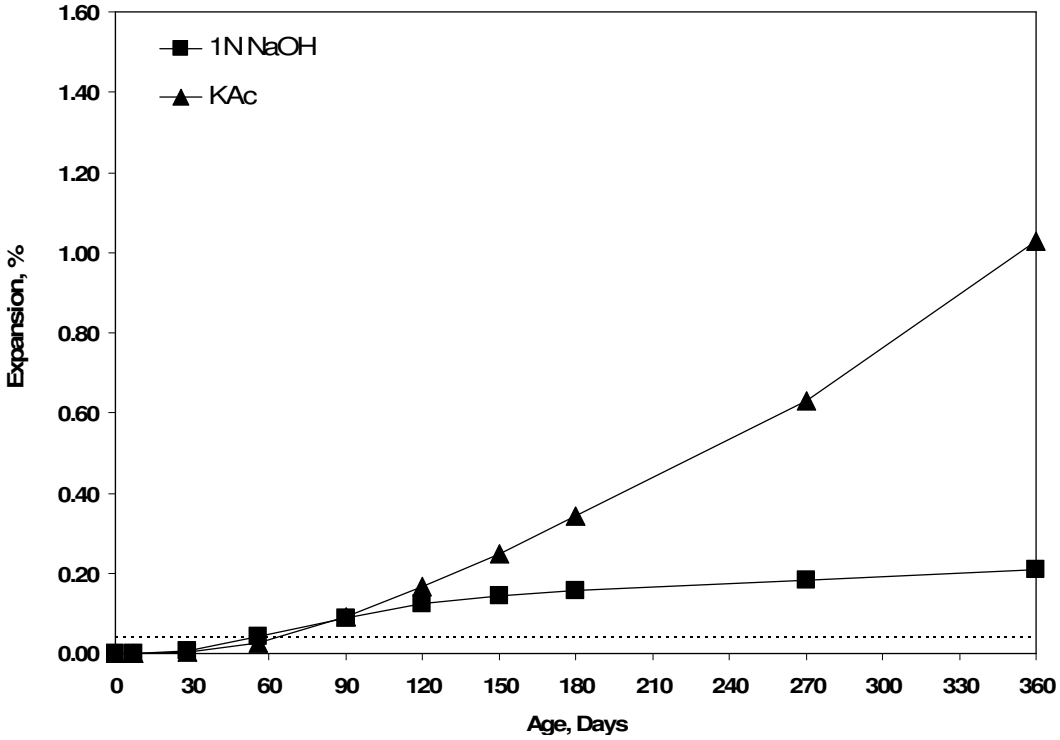

Figure 4.6: Modified ASTM C 1293 Test for the North Carolina Aggregate

From the results of the standard ASTM C 1293 tests it can be seen that at 360 days the North Carolina aggregate had the highest expansions at 0.24% expansion, with the New Mexico aggregate having the second highest expansion at 0.20%, and the South Dakota aggregate being the least reactive at 0.11%. This trend does not stay true for the modified tests with the New Mexico aggregate having the highest expansions for both the 1N sodium hydroxide and the potassium acetate. The New Mexico aggregate had expansions for the 1N sodium hydroxide and potassium acetate of 0.31% and 1.52%, respectively at 120 days. The reason for the test being cut short was due to the excessive expansions that occurred in the prisms. The North Carolina aggregate had the second highest expansions for the modified tests with 0.21% and 1.03% respectively, and the

South Dakota aggregate had expansions of 0.10% and 0.15%, respectively. Both of these being at 360 days. These results indicated that the New Mexico aggregate with an external sources of alkalis is the most reactive when compared to the other aggregates in question. This modification to the test may be a more accurate measure the reactivity of the aggregate because more than likely, there will be an external source of alkalis in the field such as deicing chemicals or other ground sources. The classification of aggregates with regard to reactivity of all the tests is listed in the appendix section D.

4.2 STANDARD AND MODIFIED ASTM C 1260 TEST (PRE-REMOVAL)

The standard and modified ASTM C 1260 (Pre-Removal) tests were to be compared to the standard and modified ASTM C 1293 tests, which is known to be the most reliable test to determine reactivity, to determine if there were any differences in results. The ASTM C 1260 procedure is a test that was created to shorten the ASTM C 1293 test, which takes at least one year, while the ASTM C 1260 procedure takes 14 days. The results of this test indicated that all of the aggregates in question where classified as potentially reactive. These results were the same for the ASTM C 1293 test, and through these two tests it confirms the reactivity of the aggregates. This is needed to compare to the standard and modified ASTM C 1260 (Post Removal) test, to determine if the removal process using hydrochloric acid affects the reactivity of the aggregate or to determine if the reactivity of the aggregate diminishes over time. The results of the tests can be seen in Figures 4.7-4.9. The same trend that was seen in the modified ASTM C 1293 tests is seen in the standard and modified ASTM C 1260 (Pre-Removal) tests, that is

the expansions at 28 days for the New Mexico aggregate in 1N sodium hydroxide and potassium acetate solutions was 1.80% and 1.59%, respectively at 16 days after casting, North Carolina had expansions of 0.76% and 0.61%, respectively, and lastly South Dakota had expansions of 0.41% and 0.44%, respectively. The results of this test will be compared to the results of the ASTM C1260 (Post Removal) tests statistically to determine if the results are significantly different or if there is insufficient data to conclude a difference. The classification of aggregates with regard to reactivity of all the tests is listed in the appendix section D.

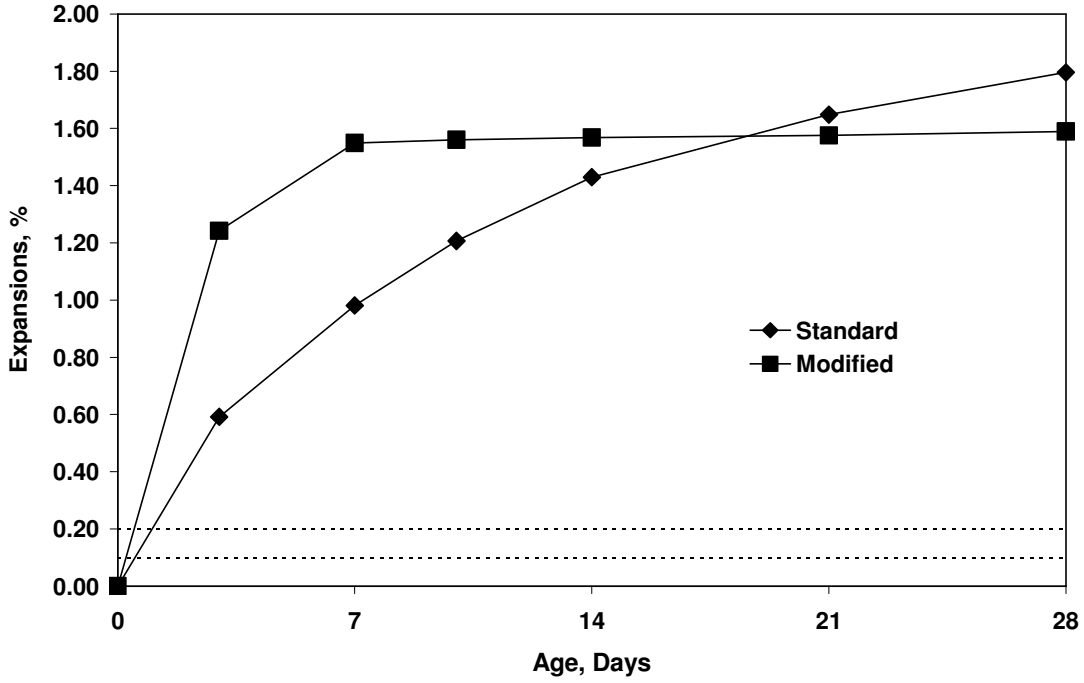

Figure 4.7: Standard and Modified ASTM C 1260 (Pre-Removal) Test for the New Mexico Aggregate

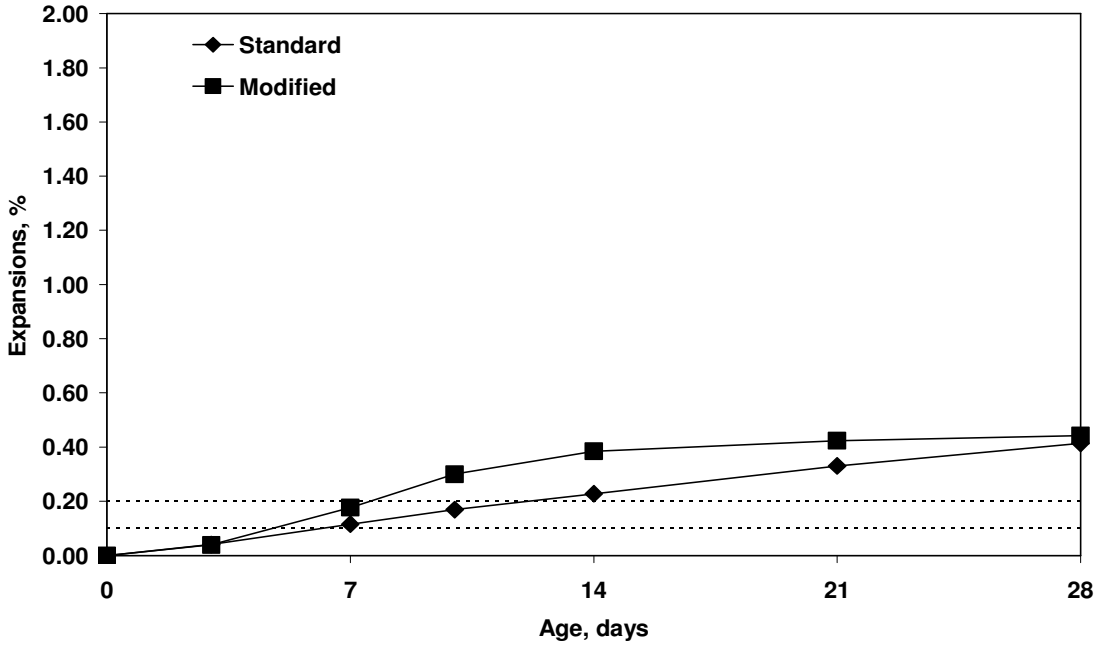

Figure 4.8: Standard and Modified ASTM C 1260 (Pre-Removal) Test for the South Dakota Aggregate

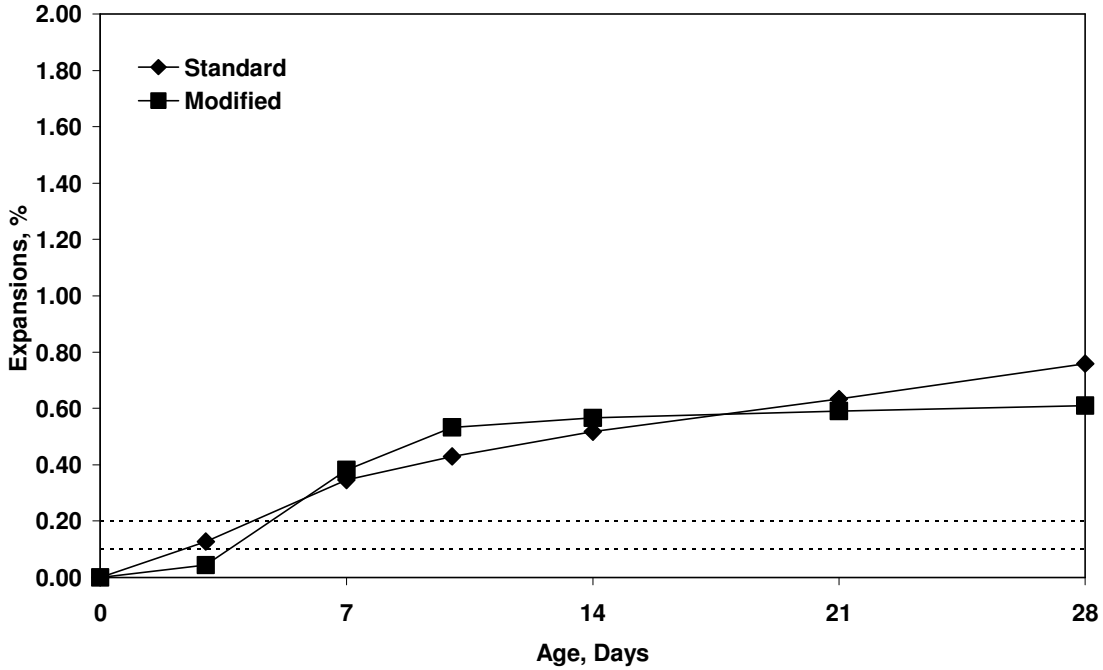

Figure 4.9: Standard and Modified ASTM C 1260 (Pre-Removal) Test for the North Carolina Aggregate

4.3 STANDARD AND MODIFIED ASTM C 1260 TEST (POST REMOVAL)

This test had two parts with the first part being the confirmation of the classifications of the known aggregates after the removal process and the second part being the application of the test to cores taken from the two airports that have known ASR problems. The first part involved the statistical comparison of the standard and modified ASTM C 1260 (Pre-Removal) test and the standard and modified ASTM C 1260 (Post Removal) test to determine any differences. There are expected to be some differences due to the fact that these tests were performed by different individuals and also the materials other than the aggregate such as the solutions and cement were of different lots that were delivered to Clemson University. The results of the four tests for each aggregate can be seen in Figures 4.10-4.15, with the explanation of the naming system being listed in Figure 3.5. From the results it can be seen that the same trend seen in modified ASTM C 1293 along with the standard and modified ASTM C 1260 tests is noticed.

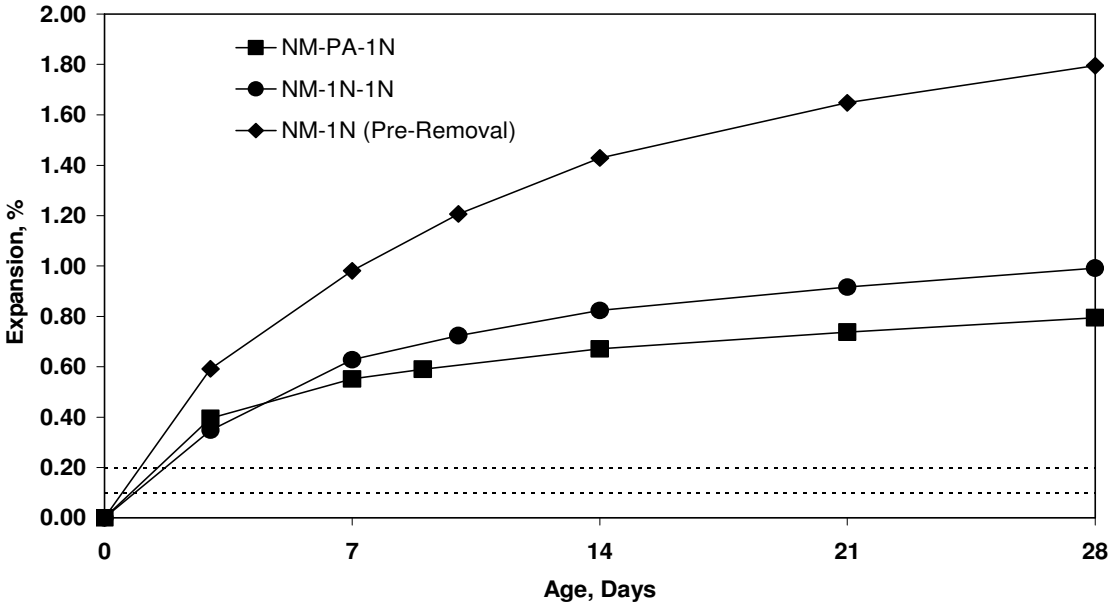

Figure 4.10: ASTM C 1260 (Pre and Post Removal) Test Results for the New Mexico Aggregate Stored in 1N NaOH

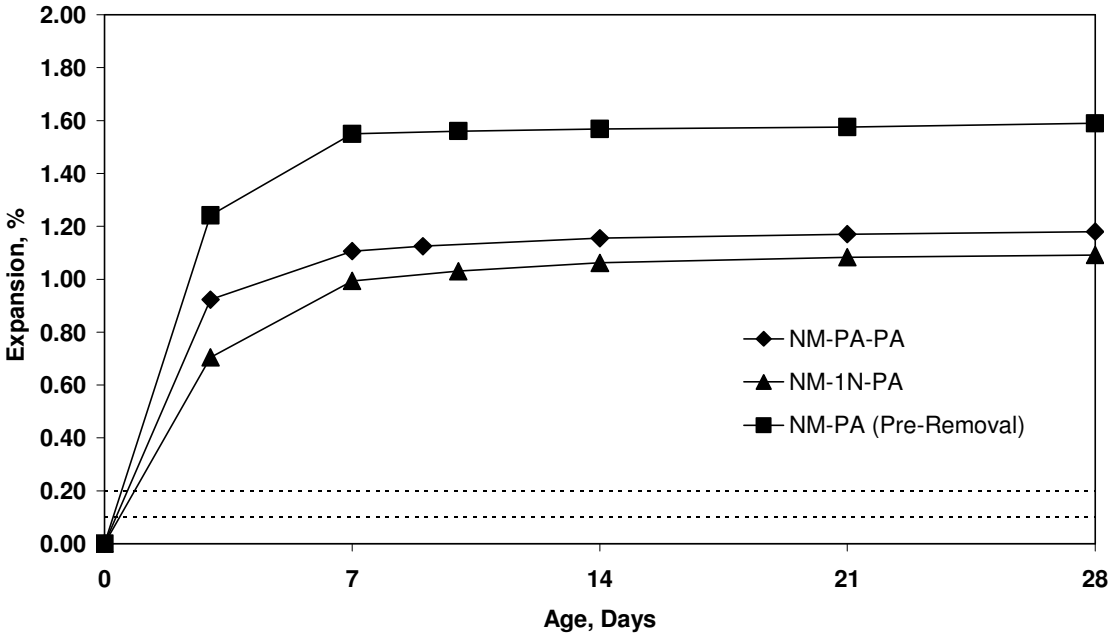

Figure 4.11: ASTM C 1260 (Pre and Post Removal) Test Results for the New Mexico Aggregate Stored in KAc

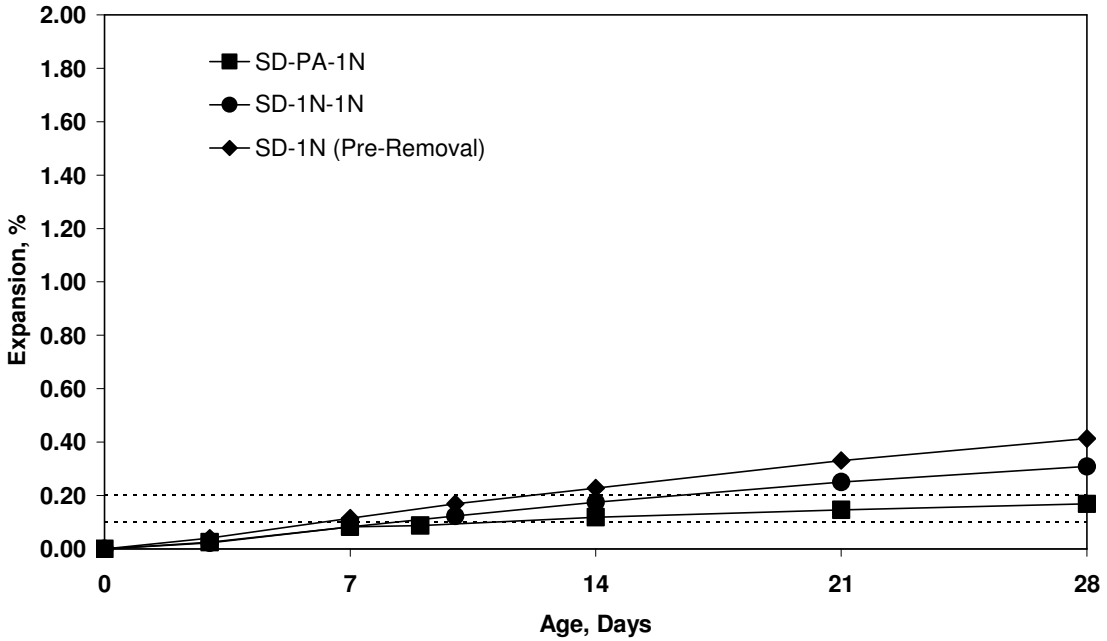

Figure 4.12: ASTM C 1260 (Pre and Post Removal) Test Results for the South Dakota Aggregate Stored in 1N NaOH

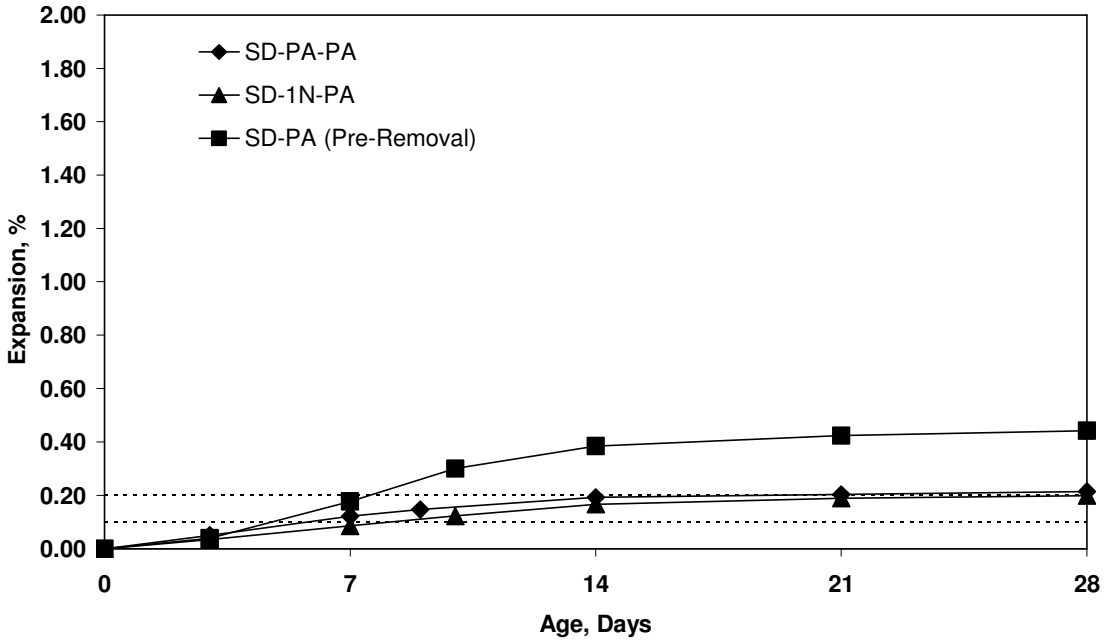

Figure 4.13: ASTM C 1260 (Pre and Post Removal) Test Results for the South Dakota Aggregate Stored in KAc

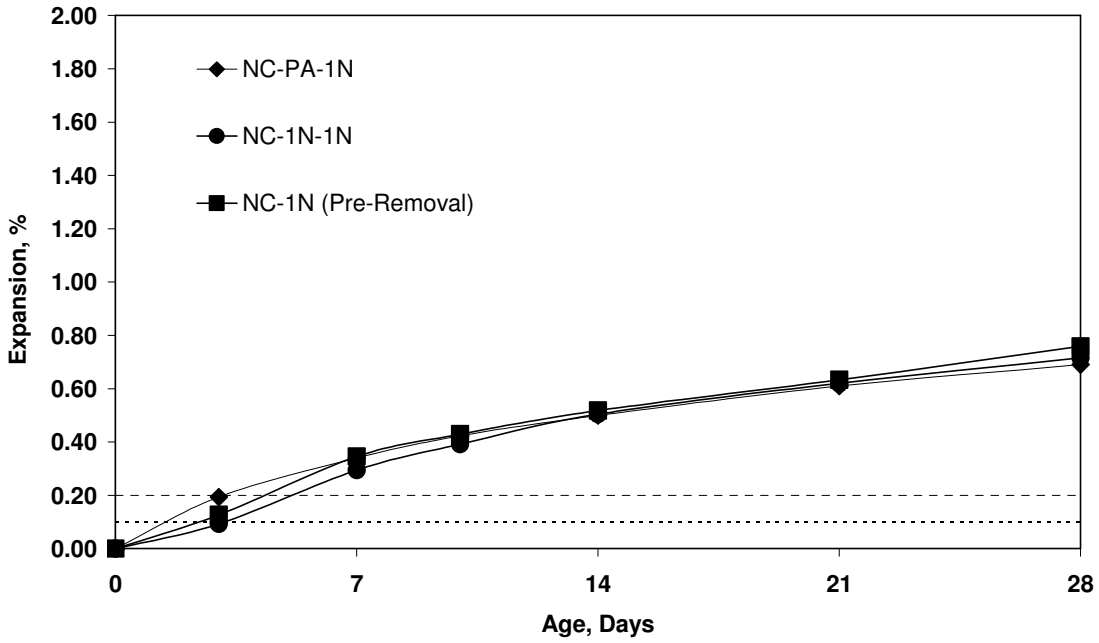

Figure 4.14: ASTM C 1260 (Pre and Post Removal) Test Results for the North Carolina Aggregate Stored in 1N NaOH

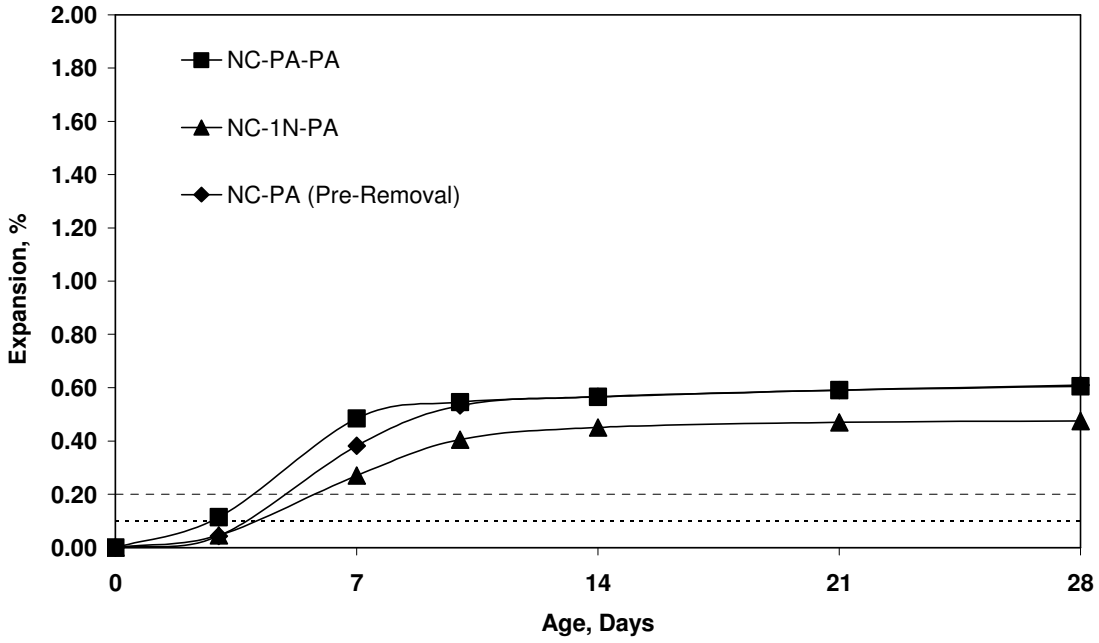

Figure 4.15: ASTM C 1260 (Pre and Post Removal) Test Results for the North Carolina Aggregate Stored in KAc

The trend of the New Mexico aggregate having the highest expansions followed by the North Carolina and South Dakota aggregate was observed. The New Mexico aggregate had a range of expansions between 1.16 and 0.67% at 16 days after casting, followed by the North Carolina aggregate having expansions between 0.57 and 0.45%, and finally the South Dakota aggregate had expansions between 0.19 and 0.12%. All of the tests for the three aggregates indicated that there may be potential for reactivity, but the South Dakota samples indicated that the aggregates may be potentially reactive or innocuous. The South Dakota samples had expansion values between 0.19 and 0.12%, which falls between the 0.1% and 0.2% limit, which indicates that the aggregate can either exhibit reactive or innocuous behavior. The statistical analysis showed that for the majority of comparisons there were significant differences in average expansions between the pre and post removal ASTM C 1260 tests, the results of these comparisons are displayed in Tables 4.1-4.3. The statistical analysis consisted of the creation of analysis of variance tables using a level of significance of 95%, or also stated as using an α value of 5%. The X's on the table indicate significant differences and the O's represent insufficient data to conclude a difference. Although there may be minor differences, the test results still indicate that the aggregates are reactive, which coincides with the previous classifications from the Pre-Removal ASTM C 1293 and ASTM C 1260 tests. A potential reason for these differences is that with the aggregate previously reacting in the ASTM C 1293 prisms, there is a reduced amount of reactive silica present in the aggregate. This inference can be made due to the fact that the expansions in the Post Removal ASTM C 1260 bars were all smaller than there Pre-Removal ASTM C 1260 bars.

Table 4.1: Summary of Statistical Analysis of New Mexico Samples

	NM-PA-PA	NM-PA-1N	NM-1N-PA	NM-1N-1N
NM-1N	X	X	X	X
NM-PA	X	X	X	X
NM-PA-PA	N/A	X	X	X
NM-PA-1N	X	N/A	X	X
NM-1N-PA	X	X	N/A	X
NM-1N-1N	X	X	X	N/A

Table 4.2: Summary of Statistical Analysis of South Dakota Samples

	SD-PA-PA	SD-PA-1N	SD-1N-PA	SD-1N-1N
SD-1N	X	X	X	X
SD-PA	X	X	X	X
SD-PA-PA	N/A	X	X	X
SD-PA-1N	X	N/A	X	X
SD-1N-PA	X	X	N/A	O
SD-1N-1N	X	X	O	N/A

Table 4.3: Summary of Statistical Analysis of North Carolina Samples

	NC-PA-PA	NC-PA-1N	NC-1N-PA	NC-1N-1N
NC-1N	X	X	X	X
NC-PA	X	X	X	X
NC-PA-PA	N/A	X	X	X
NC-PA-1N	X	N/A	O	X
NC-1N-PA	X	O	N/A	X
NC-1N-1N	X	X	X	N/A

The second part of this test involved the application of this test method to field cores taken from two specific airports with known ASR problems. The aggregate removal process was done exactly the same with these cores as with the ASTM C 1293 prisms. The results of these two tests can be seen in Figures 4.16 and 4.17.

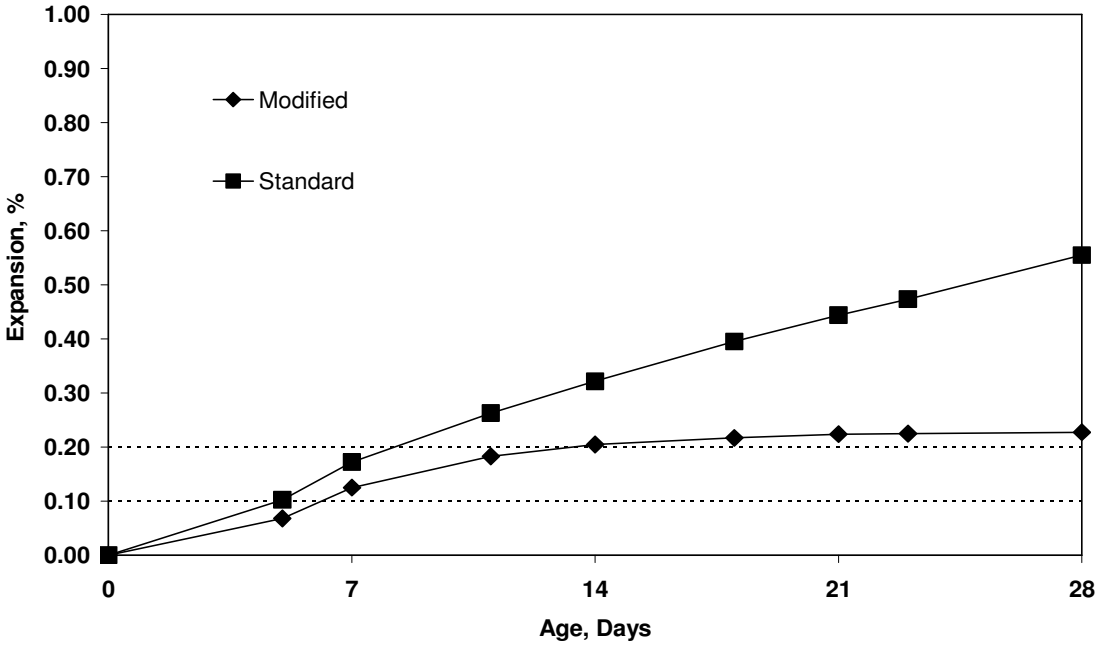

Figure 4.16: Standard and Modified ASTM C 1260 (Post Removal) Tests for the Airport #1 Aggregate

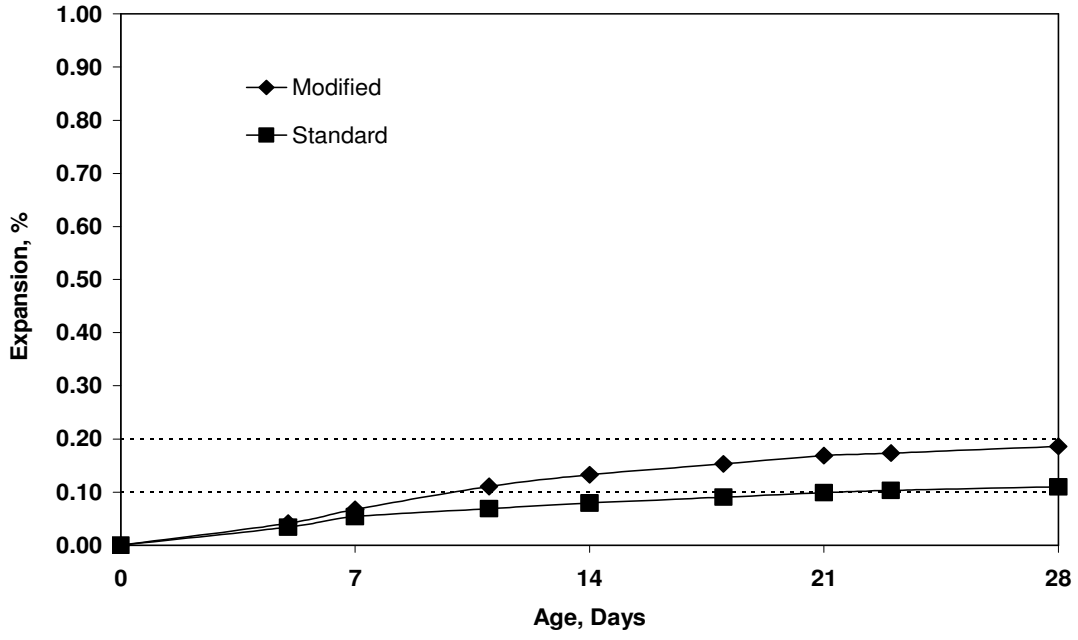

Figure 4.17: Standard and Modified ASTM C 1260 (Post Removal) Tests for Airport #2 Aggregate

The results of the test indicate that for the first airport, the aggregate was potentially reactive and for the second airport the aggregate can either exhibit reactive or innocuous behavior. The results of this test indicate that the aggregates were more than likely reactive because the reactivity of the aggregate may have diminished due to the reduced amount of reactive silica, which was assumed earlier in the calibration of the test method. The classification of aggregates with regard to reactivity of all the tests is listed in the appendix section D. All in all, this process was successful in determining the potential for reactivity in aggregates removed from a suspect pavement or runway, due to the fact that a visual inspection of the two runways in question indicated ASR problems and this was reinforced by results of the new process.

4.4 SEM AND EDX INVESTIGATION (POST REMOVAL)

It can be seen through the SEM images that there was cracking through the aggregate which is clear sign of an ASR problem. This is due to the reactive silica within the aggregate reacting with the alkalis to produce the two gels (C-N(K)-S-H and N(K)-S-H) linked to ASR, the more viscous C-N(K)-S-H gel traps the hygroscopic N(K)-S-H gel which in the presence of moisture creates expansive pressures, which cracks the aggregate. The samples looked at were the New Mexico and South Dakota samples taken from the mortar bars that were created using the aggregate removed from the ASTM C 1293 prisms that were soaked in potassium acetate. An example of the SEM images is seen in Figure 4.18. The X's on the image indicate where the EDX image was

taken and the numbers indicate which EDX image is associated with which point. The images for the reference aggregates and the two airports can be seen in the appendix section C.

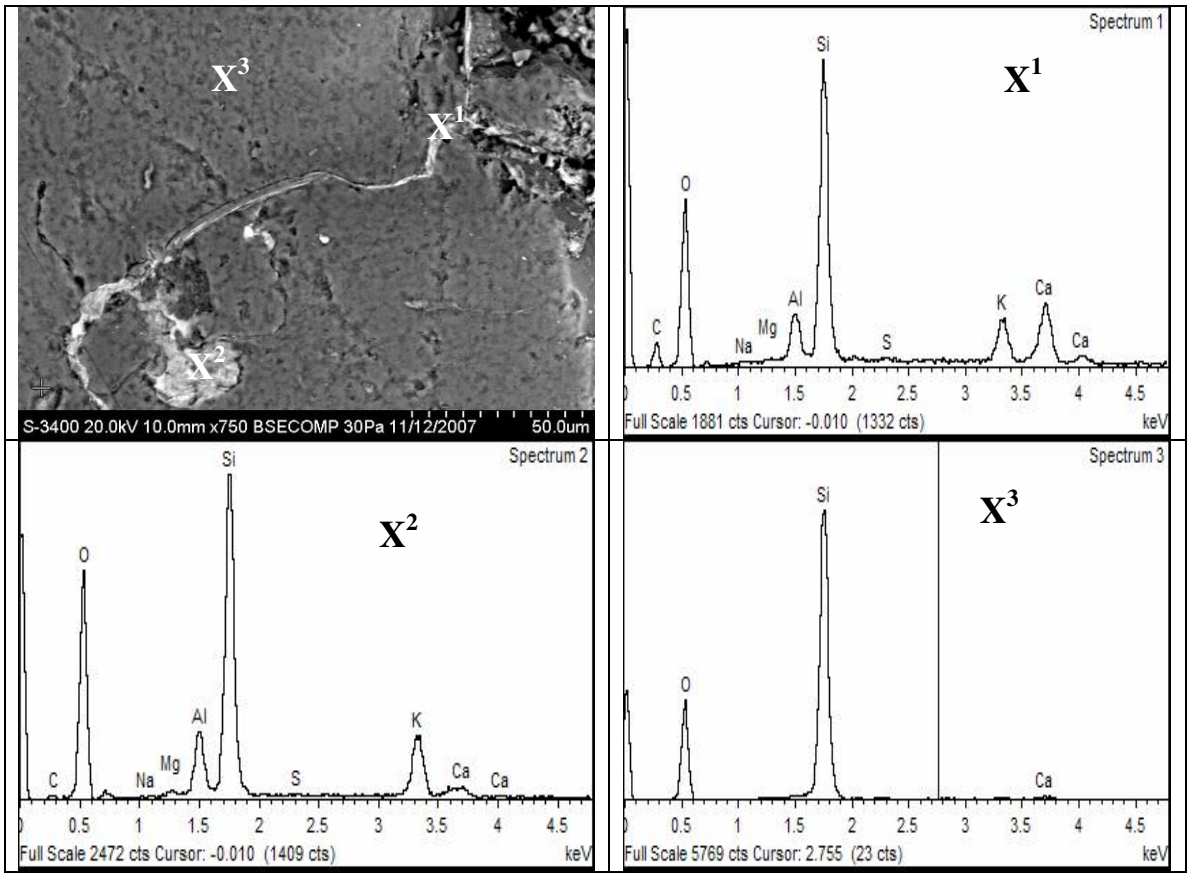

Figure 4.18: Example of the SEM and EDX Output from Mortar Bars Made with Retrieved Aggregate

5. CONCLUSIONS

Based on the tests conducted regarding the new process using existing test methods to determine the ASR potential of hardened concrete that is exposed to external alkali sources the following conclusions can be made:

- The new process using existing test methods was successful in indicating the reactivity of the reference aggregates, that is all the removed reference aggregates (New Mexico, South Dakota, and North Carolina) were determined to be reactive, which matched the results of the Pre-removal ASTM C 1293 and 1260 tests.

- The length of the removal process varies with the bonding capacity of the mortar matrix to the aggregate. A stronger bond will make the removal process last longer due to the fact that the hydrochloric acid takes longer to dissolve this bond.

- The expansions of the post-removal ASTM C 1260 mortar bars consisting of the removed aggregates tend to be lessened when compared to the initial tests consisting of the virgin aggregate. This is attributed to the reduced amount of reactive silica present in the aggregate that had reacted previously before removal.

- The post-removal ASTM C 1260 test results showed that the soak solutions had a varying effect on the expansions. There was no clear identification of which soak solution consistently produced the highest expansions.

6. RECOMMENDATIONS

6.1 RECOMMENDATIONS FROM THE CONCLUSIONS

- Use the aggregate removal process and post removal ASTM C 1260 test with both the 1N sodium hydroxide and potassium acetate to determine the worst case of expansions and use that value to predict the reactivity.

- Be aware of the fact that expansions are lessened through this process, because of the diminished reactive silica content present in the aggregate due to it being previously reacted.

6.2 RECOMMENDATIONS FOR FURTHER RESEARCH

- Testing removed aggregates that have no history of reactivity to ensure that the test method does not falsely predict reactivities.

- Try using a more accurate test method such as ASTM C 1293 on the removed aggregate to confirm the ASTM C 1260 test results performed.

- Determine the effect of the structure of the reactive silica within the aggregate. That is what effect it has on accurately predicting the reactivity of the aggregate using the new test method.

- Quantify the amount of reactive silica that was previously reacted, so that the reduced expansions from the post removal aggregate can be compensated for and lead to more accurate prediction of the reactivity.

- Testing more types of aggregates to make the test relevant to a variety of aggregates.

SECTION 2 (THE USE OF RHA TO MITIGATE ASR)

7. INTRODUCTION

7.1 PURPOSE

The purpose of this investigation is to characterize the properties of the rice husk ash (RHA) as well as to determine the ability of the RHA to mitigate ASR. Even though RHA has been incorporated into concrete production since the 1970s, and there is a limited amount of research on the subject, this line of research is being done because the RHA produced is from a new process that limits the carbon content of the ash and its effects have not been studied (25). It is anticipated that the low carbon variety of RHA will significantly improve the behavior of concrete.

7.2 BACKGROUND

RHA has been used as a pozzolanic material in concrete since the 1970s. It was originally used in limited proportions in the concrete block making industry. RHA has been used as a natural pozzolan due to its high silica content, which in turn helps to increase the pozzolanic reaction. The increased strengths resulting from the use of RHA are associated with the conversion of calcium hydroxide into a calcium-silicate-hydrate compound or C-S-H gel, which is directly related to strength gain.

Due to the increased potential of ASR caused by external alkalis such as the use of roadway/runway deicers, SCMs have been used along with admixtures such as lithium to diminish ASR potential. In this regard, RHA may have a significant potential to mitigate ASR in new concrete. RHA produced in the past contained high carbon content levels.

This resulted in unusually high admixture dosages and inconsistent quality of concrete. The properties of the low carbon RHA are not known and need to be characterized. Also, the effectiveness of this RHA in ASR mitigation is unknown. This research study aims to study the proportion of RHA and evaluate ASR mitigation.

7.3 OBJECTIVES

- To chemically and physically characterize the properties of RHA.
- To determine the reactivity of the RHA and the effects on hydration in Portland cement mortars and concrete.
- To determine the ASR mitigation potential of RHA.

7.4 SCOPE OF THE RESEARCH

The scope of research involves the physical and chemical characterization of the RHA produced by the new process, as well as the determination of reactivity of the RHA and its effects on the hydration reaction in Portland cement mortars and concrete. Once these preliminary properties are determined, the major focus of this study will be to then determine the ASR mitigation potential of RHA. The physical characterization will be done through the use of ASTM C 188, microstructure analysis through the use of an SEM with EDX analysis, surface area determination through the use of a BET with N_2 adsorption. The chemical characterization was done by x-ray fluorescence testing to determine chemical composition.

Reactivity of the RHA and its effects on the hydration process was determined through tests such as the strength activity index found in ASTM C 311 and thermogravimetric analysis of paste samples. Compressive strength testing of concretes made with RHA (ASTM C 39) was done to determine RHA's effect on compressive strength. ASTM C 642 (determination of absorption) was run on mortar samples, which was a modification to the standard, as well as concrete samples to determine the effect of RHA on permeability. Another test conducted to determine the effect of RHA on permeability was ASTM C 1202 (rapid chloride-ion permeability), which was run on mortar samples and concrete samples, the use of mortar was also a modification to the standard. SEM analyses with an EDX attachment of mortar bar samples made with RHA were looked at to determine the effect on the microscopic level. The ASR mitigation potential of RHA was determined using a standard and modified ASTM C 1567 test, which is also known as the accelerated mortar bar method to determine ASR mitigation potential through the use of pozzolans.

8. LITERATURE REVIEW

8.1 CHARACTERIZATION AND USE OF RICE HUSK ASH AS A POZZOLANIC MATERIAL IN PORTLAND CEMENT MIXTURES

8.1.1 Introduction to Rice Husk Ash (RHA)

Rice husk ash (RHA) is created through the combustion of rice husks, which is a waste product of the rice production industry. The "Rice husk [is] an agricultural waste [that] constitutes about one-fifth of the 300 million metric tons of rice produced annually in the world" (28). Currently the world production of the grain itself is about 421 million tons;

this value was reported by the Economic Research Service which is a branch of the United States Department of Agriculture (26). The rice grain is composed of two interlocking halves surrounding the rice kernel, which is the edible part. These two interlocking halves "consist of four structural layers" the layers being the outer epidermis, a layer composed of hyperderm fibers, a layer composed of spongy parenchyma cells, and finally an inner epidermis (14). A diagram of the rice grain is found in Figure 8.1.

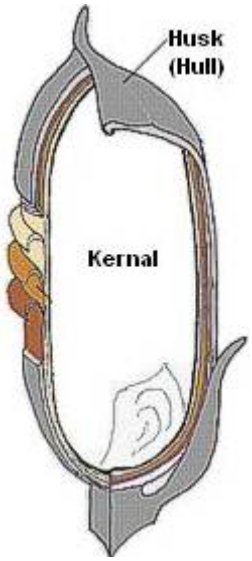

Figure 8.1: Rice Grain taken from
http://freespace.virgin.net/robmar.tin/rice/grain.jpg

The rice husk is removed by a husking process. One type process is by passes the dried out rice grain through two spinning rubber rollers, with one of the rollers spinning faster than the other. The rollers are shown in Figure 8.2.

Figure 8.2: Rice Husker taken from
http://www.sagevfoods.com/MainPages/Rice101/husker.jpg

After the husk is removed from the grain it is then incinerated and the resultant "ash is about 20% by weight of husks" (15). The degree of combustion plays a major role in the ash's potential as a secondary cementitious material. "The pretreatment, heating rate, and the time of heating affect the quantity of ash obtained" and also the "reactivity of the rice husk ash is dependent upon both its origin and its treatment" (11). It was found that "open-field burning can reduce the bulky agricultural matter to manageable amounts of ash, this has been the usual method of disposal" (14), but in the United States, a drawback is that "open heap burning of rice hulls generates undesirable atmospheric pollutants which are not permissible by USEPA" (25). Other than open heap burning, a few alternatives were proposed: use as a secondary fuel, used as animal fodder, use in the paper industry, or dumping in a landfill. It was found that "rice hulls generate 14 MJ/kg of energy" which could make it possible for use as a secondary fuel, but its cyclic availability may cause problems (25). The husks were found to have "negligible digestible protein content" which would make the material not suitable as animal feed, and the husks were found to be "high in ash and lignin contents (20-30%)", which also made it not suitable for the paper production industry (2). The final alternative proposed

was eliminated due to the husks causing “anaerobic decomposition” which would lead to the collapse of the landfill site. With the limited options of rice husk disposal, it is important to determine the effect of combustion on the rice husks to make it suitable for use as a natural pozzolan due to the fact that “gainful utilization of this agricultural product would result in increased revenue for rice producers” and it would also recycle what would otherwise be a waste product (25). Along with effects of combustion on the reactivity and chemical composition of the ash produced the effects of size of the ash particles, and RHA incorporated concrete will be discussed in this review.

8.1.2 Production of Reactive RHA

The incineration of the husk to produce the ash is a process that plays a major role in the successfulness of the use of the RHA as a secondary cementitious material. It has been found that “high temperature RHA is no better than ordinary sand as far as pozzolanic behavior is concerned” (14). Having said this, the temperature of incineration is a major role in the form of the silica that is produced. That is at higher temperatures a crystalline form of silica is produced, while at lower temperatures an amorphous form is produced. It was concluded that “incineration of rice husk in the temperature range of 550-700°C is generally found to produce amorphous silica in the ash, while temperatures in excess of 900°C produce unwanted crystalline forms” (2). The form of the silica produced from open heap burning is of low reactivity due to large temperature gradients and high temperatures producing mainly crystalline silica. The form of the silica plays a major role in the reactivity of the ash, which is the ash’s participation in the pozzolanic reaction.

If the ash is of crystalline form, then this ash will not react as well with the calcium hydroxide to produce more C-S-H gel as compared to an amorphous form, therefore, not helping to increase strength, or in other words being as effective as a pozzolanic material. When used in conjunction with lime only, the "reactivity of ash towards lime is strongly dependent on the temperature at which the ash is produced", this again confirms the effect of temperature on the RHA reactivity (12). It was also found that heating time also plays a role in the form of silica produced "heating temperature[s] low and heating time[s] short, help to produce amorphous silica" (8). The primary way to discover if the RHA is in the amorphous form or the crystalline form is to perform an x-ray diffraction analysis on the sample, therefore, this is a major test that needs to be performed to initially screen ashes to determine if they will be efficient as a pozzolan. Another property that is affected by combustion temperature and time is the color of the ash produced. It has been hypothesized that "color changes are associated with the completeness of the combustion process as well as structural transformations of the silica in the ash" and that a "white color is an indication of a complete oxidation of the carbon in the ash" (2). The typical color ranges of ashes are usually from a blacker material, due to carbon, to the white color, which has complete oxidation of the carbon (C). The carbon content of the ash also plays a role in the RHA usage, because an ash with high carbon content can not be used efficiently as a secondary cementitious material. Two processes have been used to produce amorphous silica in the ash; these processes are the fluidized bed and fixed bed method. Neither of these processes "has successfully produced commercial-scale off-white amorphous SiO_2 with <1% amorphous C" (25). A new process has been

proposed to limit the carbon contents and this process uses template free ZSM-5 from siliceous rice hull ash, but this process is still in the research stage (25). The amount of silica available in the ash is also related to the combustion temperature, it was found that the "higher the burning temperature, the greater the percentage of silica in the ash" (18). Typical values of silica range from about eighty to ninety-five percent, with the more reactive ashes having the higher silica contents around ninety-five percent. It has been found that the rice plant absorbs the silica from the soil, so the soil the rice is grown in also plays a large role in its effectiveness as a pozzolan. The alkali content of the ash is usually in the forms of potassium oxide (K_2O) and sodium oxide (Na_2O) with the typical range of alkalis from one to five percent, with potassium oxide being the main alkali. Typical potassium oxide ranges can also be from one to five percent, with it being found that "relatively high K_2O content originates mainly from fertilizers" that were used in the farming of the rice (3). This potassium oxide also plays a role because the "K_2O content is enough to accelerate the sintering and crystallization of the ash" (8). This is another indication of the importance of soil type and how any alterations to the soil such as a fertilizer have on the chemical composition. A final effect of the amount of potassium is that the "pH value increases due primarily to the potassium in RHA dissolving in water" this pH increase caused by the RHA can also be helpful in the corrosion protection of the steel (11). This is directly due to a phenomenon that occurs at a pH around nine, where a passivity layer forms around the steel to help protect against mass loss due to corrosion. All of these factors revolve around the combustion duration and temperature of the rice

husk, which means that an optimal process needs to be found to take into account the temperature, duration, carbon contents, silica contents, and alkali contents.

8.1.3 Size Affect of RHA on Reactivity

Along with the content of silica in the ash, the size and surface area of the ash particles play a large role in the reactivity of the ash, because the "reactivity of RHA is attributed to its high content of amorphous silica, and to its very large surface area governed by the porous structure of the particles" (4). The rice hull after combustion is very vesicular and, therefore, creates large surface areas, but at a "certain stage of grinding the RHA, the porous structure of the particles will collapse, thereby reducing the surface area of the RHA" (4). The typical way of grinding is with the use of a vibrating ball mill, an example is shown below.

Figure 8.3: Vibrating Ball Mill from Gold Supplier
http://img.alibaba.com/photo/50509510/Ball_Mill.jpg

Typical surface areas reported by the BET method as N_2-adsorption are between 30-100 m^2/g, and "Mehta has argued that grinding of RHA to a high degree of fineness should be avoided, since it derives its pozzolanic activity mainly from the internal surface area of

the particles" (4). Along with the effect of the pozzolanic reaction, there is a secondary effect called the filler effect, which is created due to the increased fineness of the RHA. It has been found that "prolongation of pulverizing time does not necessarily increase the specific surface area. The data shows the lowest specific surface area at longer pulverizing times, due to secondary agglomeration of fine powders"; this also shows how the grinding time is an important factor in creating a strong pozzolanic material (23). This filler effect is more pronounced when a coarser cement is used such as a Type I cement. This is mainly due to a more well graded particle size distribution, as compared to using a Type III cement which has smaller grains creating a more uniformly graded material. It was found that "blending finely ground RHA with such a coarser cement, higher packing densities near the aggregate grain interface can be expected" that is the weakest part of the concrete matrix, the bond between the cement matrix and the aggregates also known as the interfacial transition zone (ITZ), is strengthened due to the increased density (4). The ITZ is known to have a higher water to cement ratio compared to the surrounding cement matrix, therefore, leading to decreased strengths. A secondary effect of the RHA to increase the performance of the ITZ is the ability of the ash particles to absorb the water in the ITZ, therefore, reducing the water to cement ratio and in turn creating a stronger bond. The actual size of the ash particles can range from 5-100 μm and the "maximum activity has been observed in case of RHA less than 45 μm" (1). The grinding time of the RHA is almost as important as the combustion parameters and also needs to be taken into account when determining the efficiency of RHA as a natural pozzolan.

8.1.4 ASR Mitigation Potential

The incorporation of a high quality RHA in concrete will help to reduce a bulk waste along with improving physical characteristics of Portland cement concrete. These characteristics may include: increased durability, decreased chloride-ion permeability, increased strengths, and reducing adiabatic temperature rises. It has been seen that the durability has been increased as indicated by lower expansion in mortar bars in the ASTM C 1567 procedure (Accelerated Mortar Bar Method). "Cements containing 10% rice husk ash have been shown to be quite effective in combating expansion due to alkali-aggregate reaction" (14). It was also found that "the degree of damage of the aggregate decreased markedly when the level of the RHA replacement was increased," which also confirms the decreased expansions due to the fact that less alkalis from the aggregate are reacted and, therefore, less ASR gel is formed resulting in decreased expansions (9). Further research should be done with an optimized RHA, with respect to size, surface area, silica form and quantity.

8.1.5 RHA Incorporation Effect on Chloride Ion Permeability

To determine the chloride-ion permeability, an ASTM C 1202 test must be performed. Typical values for normal Portland cement concrete are around 2500 coulombs, which is considered moderate penetrability, but "RHA concretes showed low to very low chloride-ion permeability (600-1210 coulombs)" (3). This increased resistance to chloride-ion permeability due to the incorporation of RHA helps to limit the chloride content in the concrete, and in the case of reinforced concrete, this reduction in chloride ion contents helps to increase the reinforcing steel's resistance to corrosion.

8.1.6 RHA Incorporation Effect on Strength

Increased strengths have been seen in RHA incorporated concrete compared to a control Portland cement concrete. This is directly due to the calcium hydroxide presence or Portlandite in the cement matrix, "therefore, in comparison to the concrete without RHA addition, there will be more C-S-H gel and less Portlandite in the concrete with RHA addition" (26). This increase in C-S-H gel is completely due to the pozzolanic reaction occurring between the amorphous silica and the excess calcium hydroxide producing the C-S-H gel. The increased strength is also due to the refinement of the ITZ, which was discussed earlier.

8.1.7 RHA Incorporation Effect on Heat of Hydration

It has been determined that concrete incorporating RHA can "reduce adiabatic temperature rise compared to normal PCC" (16). This is of major importance in mass concrete pours that is concrete pours greater than $1m^3$, due to large temperature gradients created in the mass pour. This large temperature gradient can lead to a phenomenon called delayed ettringite formation (DEF) or internal sulfate attack, which causes the creation of ettringite which is an expansive product, which leads to the deterioration of the concrete structure. This reduced adiabatic temperature rise caused by the incorporation of RHA has the potential to reduce costs associated with cooling the concrete structure. This reduced temperature rise is not an indication of reduced strengths compared to normal PCC as seen earlier. As pozzolans come, the closest comparison of RHA to any other is with silica fume. It has been found that RHA has 1.5 times the surface area of silica fume. The time to reach maximum temperature in

concrete made with silica fume compared to RHA is shorter, which "indicates high reactivity for the RHA and its effect on the acceleration of cement hydration due to its high specific surface" (28). There are, however, some downsides to the use of RHA in PCC and they include increased water demand, increased admixture demand, increased final set times, and increased drying shrinkage.

8.1.8 RHA Incorporation Effect on Concrete Proportioning

With the use of RHA in Portland cement concrete there are certain precautions that need to be addressed. The water demand on average was about 104% and this was mainly due to "absorbed water in the great number of mesopores existing in RHA particles" (26). Increases in air entraining admixtures and super plasticizer admixture dosages are noticed and these dosages increase with the increase in RHA content. For RHA's with high carbon content the "RHA increases dosage of air entraining admixture due to the carbon in the RHA" (3). Increased drying shrinkage can be associated with the increased water demand of the RHA. However, if sufficient curing is in place this can be avoided. A final concern in proportioning RHA in PCC is the reduced specific gravity of RHA, which is typically in the range of 2.04-2.20. This may cause problems when replacing cement due to the fact that Portland cement has a specific gravity around 3.15. This large difference in specific gravities may make it difficult to replace the cement by mass, because a mass of RHA takes up a lot more volume than an equivalent mass of Portland cement. This may make it more useful for a volumetric replacement instead of a mass replacement.

8.1.9 Conclusion

This review focuses mainly on the effect of combustion time and temperature range on the reactivity of the RHA, the effect of grinding time on reactivity of RHA, and finally the effects on the physical characteristics of PCC incorporating RHA. It has been shown that excessive temperatures above 800°C and longer combustion durations help to create mainly crystalline silica, while temperatures in the range of 500-700°C for shorter durations create mainly amorphous silica, which is what is needed for an RHA to be an effective pozzolan. The grinding time affects the surface area of the ash particles produced and the typical surface areas being reported by the BET method as N_2-absorption was between 30-100 m^2/g. It was found that excessive grinding actually can reduce surface area, due to the breaking down of the cellular structure of the ash particles, and for RHA to be reactive the ash particles need a higher surface area. There are positives and negatives to the incorporation of RHA in PCC, with the positives outweighing the negatives. The positives include increased durability, decreased chloride-ion permeability, increased strengths, and reducing adiabatic temperature rises, which all have there individual merits. The negative effects mainly affect the cost of materials such as increased admixture dosage or increased curing costs, but these increased costs should be able to be overcome with the cement replacement or in the case of mass concrete pours with the decreased cost of cooling the concrete. With all the information presented the keys to producing a valuable pozzolanic material from a bulk waste and incorporating into a good performing concrete is to take into account the

combustion time and duration, the grinding time, and finally the proportioning of the PCC with RHA.

The first part of this study being to determine the ASR potential of hardened concrete makes the second part of the study an obvious next step. Due to increased usage of potassium acetate based deicers and anti-icers, the potential for ASR problems has been increased, which leads to the need for a successful ASR mitigator. Determining a successful ASR mitigator would help to prevent the first step from being necessary. This review indicated that the limited available research concluded the addition of RHA was successful in ASR mitigation, which is one of the two reasons why this material was included in this two part study. The fact that this material is produced through a new process to limit the carbon contents is the second reason for its selection in this study. The combination of the limited available research on the subject indicating RHA as an ASR mitigating material and the fact that this material is created through a new process makes RHA a good candidate for this study.

9. MATERIALS AND METHODS

9.1 TEST MATERIALS

The Rice husk ash used in this research was provided by ChK Group Inc. of Plano, TX. The rice hulls were obtained by ChK Group Inc. from the Busch Agricultural Resources, Inc. out of St. Louis, MO, which is a subsidiary of Anheuser Busch, Inc. These hulls were then ground and passed through a 400-mesh sieve, then heated in a muffle furnace at 500°C for 4 hours. This produced an ash with a surface area of 35 m^2/g, the chemical composition presented in Table 9.1 determined by X-ray fluorescence analysis, and the particle size distribution presented in Table 9.2. With all of the preceding information being provided by the ChK Group Inc.

Table 9.1: Received RHA Chemical Composition

Elemental Composition	**Percentage**
SiO_2	94.80
Al_2O_3	0.52
K_2O & Na_2O	2.92
Carbon	0.24
P_2O_5	1.09
Fe_2O_3	0.13
MnO	0.39
Total	100

Table 9.2: Received RHA Size Distribution

Particle Size	**Percentage Retained**
<10µm	2
15-30µm	22
30-90µm	76
Total	100

The cement used in all of the tests was a high-alkali Type I cement with the chemical composition listed in Table 10.3. The fine aggregates used in all of the mortar tests was silica sand produced by the U.S. Silica Company and, for the ASTM C 1567 test, a crushed limestone with known reactivity was used. For the concrete tests, natural sand was used from a local source.

Table 9.3: High-alkali Type I Chemical Composition

Oxide	%
SiO_2	19.74
Al_2O_3	4.98
Fe_2O_3	3.13
CaO	61.84
MgO	2.54
SO_3	4.15
LOI	1.90
$Na_2O_{equivalent}$	0.82
K_2O	0.84
Na_2O	0.27
Insoluble Residue	0.25
C_3A	8.97
C_3S	46.6
Autoclave Expansion	0.12

9.2 TEST METHODS

9.2.1 X-Ray Fluorescence Analysis

X-Ray fluorescence analysis was conducted by the ChK Group Inc. to determine the chemical composition of the RHA produced through their new process. The samples tested were taken after the rice hulls were heated at 500°C for 4 hrs. The analysis was done using an Oxford Instruments energy dispersive X-ray fluorescence spectrometer, model ED-2000.

9.2.2 Surface Area Determination

Surface area determination was conducted by the ChK Group Inc. This test was conducted on the RHA after 4 hours of heating at 500°C. This test was conducted using an analyzer that uses the BET method. This apparatus used uses nitrogen gas adsorption to determine surface area.

9.2.3 ASTM C 188 (Density)

The test method used to determine the density of the RHA was the *Standard Test Method for Density of Hydraulic Cement*. This method was followed except for the sample size, the sample size had to be reduced due to the fact that RHA has a much lower specific gravity than Portland cement (RHA has a specific gravity around 2.08 whereas Portland cements specific gravity is 3.15). The sample size recommended was 64g and the reduced sample size was 35g. The liquid used in the test was kerosene, which met the requirement of having a density greater than 0.73 g/mL. The density was determined by

dividing the mass of the sample by the difference between the final and initial readings taken from the Le Chateliers flask.

9.2.4 ASTM C 311 (Strength Activity Index)

The determination of the Strength Activity Index is outlined in the ASTM C 311 test method entitled *Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete*. This method was used to determine the Pozzolanic Activity Index also known as the Strength Activity Index, which is an indication of the RHA's reactivity and its influence on strength gain when used as a cement replacement compared to a control Portland cement mortar. The test method was followed using the mixture proportions given in the standard, but instead of only replacing 20% of the mass of the Portland cement with the RHA, tests were performed at 5, 10, 15, and 20% mass replacement. The Pozzolanic Activity Index value was taken as the average compressive strength of the test mixture divided by the average compressive strength of the control mixture multiplied by one hundred to give a percentage. This test was run twice, with the difference being an addition of a superplasticizer. The use of the superplasticizer was due to the fact that the RHA greatly increases the water demand, which when the W/C ratio is kept constant, the mixtures with increased contents of RHA do not have sufficient water to fully hydrate the cement grains. This leads to decreased strengths that are not a direct effect of the use of RHA, but rather an indirect effect. Eliminating this effect can truly show the reactivity of the RHA and its influence of the pozzolanic reaction. The amount of superplasticizer used was dictated by the flow of the

control sample made without RHA. The superplasticizer used was Glenium 3030 NS made by BASF Admixtures Inc.

9.2.5 ASTM C 39 (Compressive Strength Determination of Concrete Made with RHA)

The compressive strength was determined by using 4"x 8" cylinder samples prepared using a control sample and a 10% by mass replacement of Portland cement with RHA. Tests were preformed at 7 and 28 days, three samples were tested for each of the two concretes at each of the test dates. The cylinders were made according to the standard by casting the specimens in two layers, rodding each of the layers 25 times with the properly sized rod. The two mix designs can be found in the appendix section H.

9.2.6 Thermogravimetric Analysis of Pastes Made with RHA

Thermogravimetric analysis was done on paste samples made with the following RHA mass replacement levels: 0, 5, 10, 15, and 20%. A superplasticizer was used to achieve matching consistencies between the samples made with RHA and the control. The starting water to cement ratio for the paste samples was a 0.45, but this changed for the samples containing superplasticizer. The cement used in this analysis was a high-alkali Type I Portland cement. The thermogravimetric analyzer used was a TGA/SDTA 851 measuring module produced by the Mettler Toledo Company. The heating rate was 10°C/min with a starting temperature range of 100-200 °C and a final temperature range of 600-700 °C. The main focus of this study was to determine the calcium hydroxide drop between 430 and 450 °C.

9.2.7 Microstructure Analysis Using an SEM and EDX Analysis of the RHA Grains Alone

Scanning Electron Microscopy (SEM) analysis was conducted at Clemson University's Electron Microscope Facility. The microscope used was a variable pressure Hitachi S-3400 SEM with an energy dispersive X-ray spectroscopy (EDX) attachment, with the EDX attachment being used to determine chemical composition in relative amounts. Samples analyzed were of the RHA alone. Samples were prepared by penetrating a two-part epoxy into a RHA powder sample until no air bubbles were visibly coming to the surface of the samples by use of a vacuum. The vacuum penetration was done using a desicator and a vacuum pump. The samples were then placed in an oven at 60℃ until the samples hardened. Once the samples were hardened, they were removed from the molds and then sliced to expose the RHA. The samples were then polished to remove surface defects. These samples were then ready to be analyzed using the SEM with EDX attachment. RHA was also looked at directly using the SEM, which means no epoxy embedding. This was done by placing the RHA in powder form onto a two sided piece of carbon tape, and then attaching this piece of tape to a platform or "stage" which was then placed in the SEM.

9.2.8 ASTM C 1202 (Rapid Chloride Ion Permeability to Determine Permeability of Concrete Made with RHA)

The determination of the rapid chloride ion permeability was done to determine the relative permeability of the concrete. The samples were made by casting 4"x 8" cylinders and then cutting the samples into 2" thick discs discarding the top and bottom 1". The samples were conditioned according to the ASTM standard, making sure that the

4 in. diameter by 2 in. thick disc samples were fully saturated at the beginning of the test. The solutions used in the test were the 0.3N NaOH solution and the 3.0% by mass NaCl solution. For the test, a control sample and a 10% (mass replacement) RHA sample were made, two samples for each were tested. The testing equipment met all the requirements of the standard.

9.2.9 Modified ASTM C 1202 (Rapid Chloride Ion Permeability to Determine Permeability of Mortar Made with RHA)

This determination of the rapid chloride ion permeability was modified by using the same dimension samples, but this time instead of concrete, mortar was used. This was done to try and determine the effect of replacement of Portland cement with RHA on the permeability of the mortar. For this test, mortar samples were made with 0% (control), 5%, 10%, 15%, and 20% RHA replacement. This replacement was all done on a mass basis. Tests were done at 14 and 28 days, with two samples tested for each replacement level at each of the respective ages. The samples were cast and conditioned in the same manner as the samples in the concrete ASTM C 1202 test and the solutions were also prepared in the same manner. The same testing equipment was used during the mortar samples as the concrete samples.

9.2.10 ASTM C 642 (Determination of Absorption of Concrete Made with RHA)

This test method was used to determine the absorption of a control concrete sample compared to a 10% by mass replacement of RHA concrete sample. One minor change to the standard was the order of the measurements taken, instead of first oven drying the

samples, the oven dry weights were taken at the end of the test to avoid the step of vacuuming the samples, because it was assumed the samples were fully saturated with water due to the fact that they were in a moisture room from the day of casting until the time of the test. In this test samples are first weighed saturated, then boiled for 5 hrs. The samples were then taken out and stored in water at 25°C for not less than 14 hrs. The samples were then reweighed and then a submerged weight of the samples was taken, the samples were then placed in the oven at 110°C for 24 hrs and then weighed dry. The samples for this test were conditioned the same way as the samples for the ASTM C 1202 test, making sure that the 4" diameter by 2" thick samples were at saturated condition prior to the boiling of the samples.

9.2.11 Modified ASTM C 642 (Determination of Absorption of Mortar Made with RHA)

The only modification to this test was the use of mortar instead of concrete, as for the rest of the test it was conducted following the ASTM standard except for the same minor change discussed in the standard ASTM C 642 section. The reasoning for this was also to determine the effect of replacing Portland cement with RHA on the permeability of the samples. The samples were of the same mixes prepared for the modified ASTM C 1202 test, having samples of 0% (Control), 5%, 10%, 15%, and 20% mass replacement of RHA. The conditioning was the same as for the standard ASTM C 642 test conducted, making sure that the samples were in the fully saturated condition at the beginning of the test.

9.2.12 Standard and Modified ASTM C 1567 (Accelerated Mortar Bar Method)

The determination of the mitigation potential of ASR through the use of RHA was determined through the use of ASTM C 1567 or the accelerated mortar-bar method. The test was done in a standard format along with a modified format, with the modification being the use of potassium acetate, a deicer, instead of the standard 1N NaOH solution. The mass replacement levels of RHA used in this test were 0, 5, 10, 15, and 20%. The fine aggregate used in the size fractions indicated in the test method was a crushed limestone.

9.2.13 SEM Analysis of the Mortar Bar Samples

SEM analysis was conducted at Clemson University's Electron Microscope Facility. The microscope used was a variable pressure Hitachi S-3400 SEM with an energy dispersive x-ray spectroscopy (EDX) attachment, with the EDX attachment being used to determine chemical composition in relative amounts. Samples looked at were of the mortar bar sections taken from the ASTM C 1567 samples. Samples were prepared by vacuum penetrating a two-part epoxy into the sectioned mortar bar samples until no air bubbles were visibly coming to the surface of the samples. The vacuum penetrating was done using a desicator and a vacuum pump. The samples were then placed in an oven at 60℃ until the samples hardened. Once the samples were hardened they were removed from the molds and then sliced to expose the mortar bar section surface. The samples were then polished to remove surface defects. These samples were then ready to be analyzed using the SEM with EDX attachment.

10. RESULTS AND DISCUSSION

10.1 INITIAL STUDIES BY THE ChK GROUP INC.

The first properties tested of the RHA produced by the ChK Group Inc. were the chemical composition and the surface area determination. Both of these tests were conducted by the ChK Group Inc. before the RHA was delivered to Clemson University. Table 10.1 shows the results of the x-ray fluorescence analysis which is used for determination of chemical composition. The very high silica content (~95%) is an early indication that the RHA will promote a strong pozzolanic reaction and the low carbon content (~0.24%) is an indication that the process created by the ChK Group was successful in decreasing the carbon content to negligible amounts. The surface area determination was also conducted by the ChK Group and was done using the BET method with nitrogen adsorption. The value was about 35 m^2/g, which shows that the RHA has a very high surface area, which leads to increased water demands in mortar and concrete incorporating RHA but also leads to increased reactivity of the RHA, which in turn increases the pozzolanic reaction.

10.2 DENSITY

The first characteristic of the RHA tested at Clemson University was the density of the RHA, which was determined using ASTM C 188. The test had to be slightly altered due to the fact that RHA has a low specific gravity. The alteration being the reduction of the sample size. The specific gravity was found to be 2.08, which is much lower than Portland cement (SG = 3.15). This indicates that a mass replacement of RHA for

Portland cement may not be the best method of proportioning mortar or concrete, but a volumetric replacement may be the better alternative.

Table 10 .1: Received RHA Chemical Composition

Elemental Composition	**Percentage**
SiO_2	94.80
Al_2O_3	0.52
Alkalies	2.92
Carbon	0.24
P_2O_5	1.09
Fe_2O_3	0.13
MnO	0.39
Total	100

10.3 STRENGTH ACTIVITY INDEX

To determine the level of strength development when used with Portland cement in concrete, or the effectiveness of converting calcium hydroxide into C-S-H gel, also known as the pozzolanic reaction, ASTM C 311 was used. The value obtained through this test is known as the strength activity index (SAI) or pozzolanic activity index. The values of RHA mass replacement used were 0, 5, 10, 15, and 20%. This test was run twice, once using superplasticizer and once without. The reasoning behind this is listed in section 9.2.14. The results of the first test showed that with increasing RHA contents, the strength activity index increased at 5% replacement, but with higher levels of

replacement, a SAI of less than 100% was seen indicating a detriment to strength gain. These results were not the same for the second set of tests using the superplasticizer. The values of SAI were all above 100% indicating that a pozzolanic reaction was occurring, which lead to higher strengths than the control. The results of these two tests are displayed in Figure 10.1 with the strength activity index on the y-axis and the replacement level on the x-axis. The actual strength results are shown in Figure 10.2. The use of the superplasticizer helped to decrease the effect of increased water demand caused by the RHA, which can lead to the decreased amount of cement grains being hydrated. The decreased amount of cement grains being hydrated leads to lower strengths, which would explain the reduced strengths of the first test without superplasticizer. Overall, the SAI values of the second tests are a better description of the effectiveness of RHA in increasing compressive strengths or in actuality the effectiveness of the pozzolanic reaction in creating more C-S-H gel, which is responsible for strength gain.

10.4 CONCRETE STRENGTH COMPARISON

One limitation of this standard is these tests are done with mortar which may not provide a direct correlation to the strength of concrete. Due to this concern, concrete was made with RHA, but due to material restrictions only control samples and 10% by mass replacement of Portland cement with RHA samples were made. The 10% by mass replacement was chosen from the results of the SAI test with superplasticizer, because it had the highest SAI value. These concrete samples were tested at 7 and 28 days of age.

At both ages, the strength values were greater for the 10% RHA replacement. This again is a confirmation that a pozzolanic reaction is occurring to increase the amount of C-S-H gel to in turn increase the compressive strength. The results of this data is displayed in Figure 10.3.

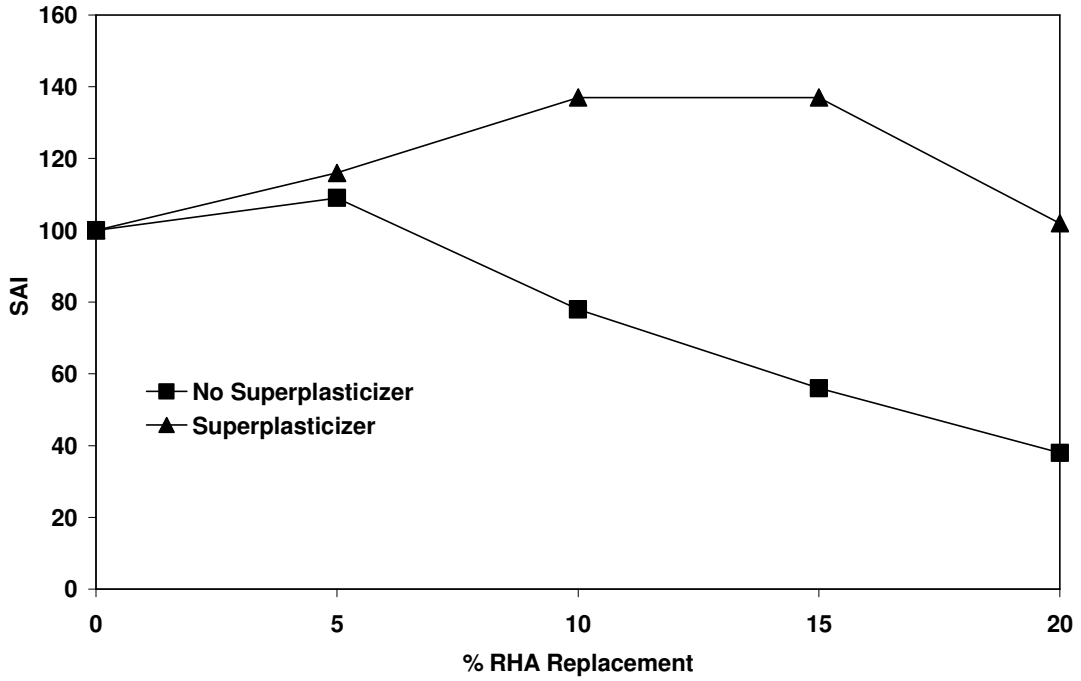

Figure 10.1: Strength Activity Index versus RHA replacement level

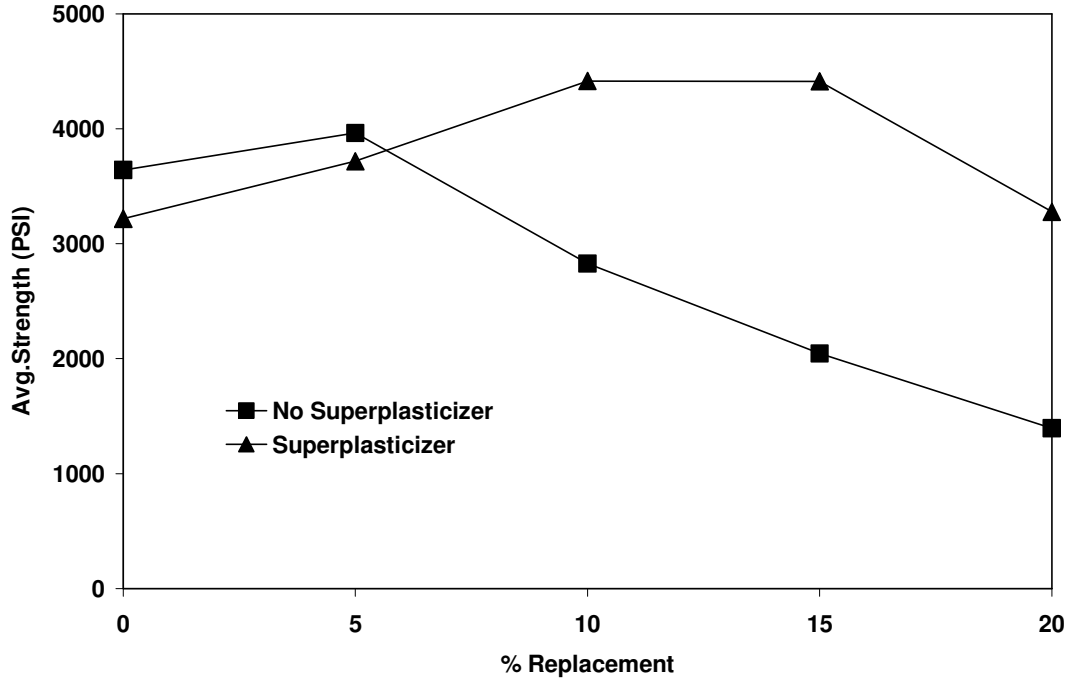

Figure 10.2: Avg. strength versus RHA replacement level for the Strength Activity Index test

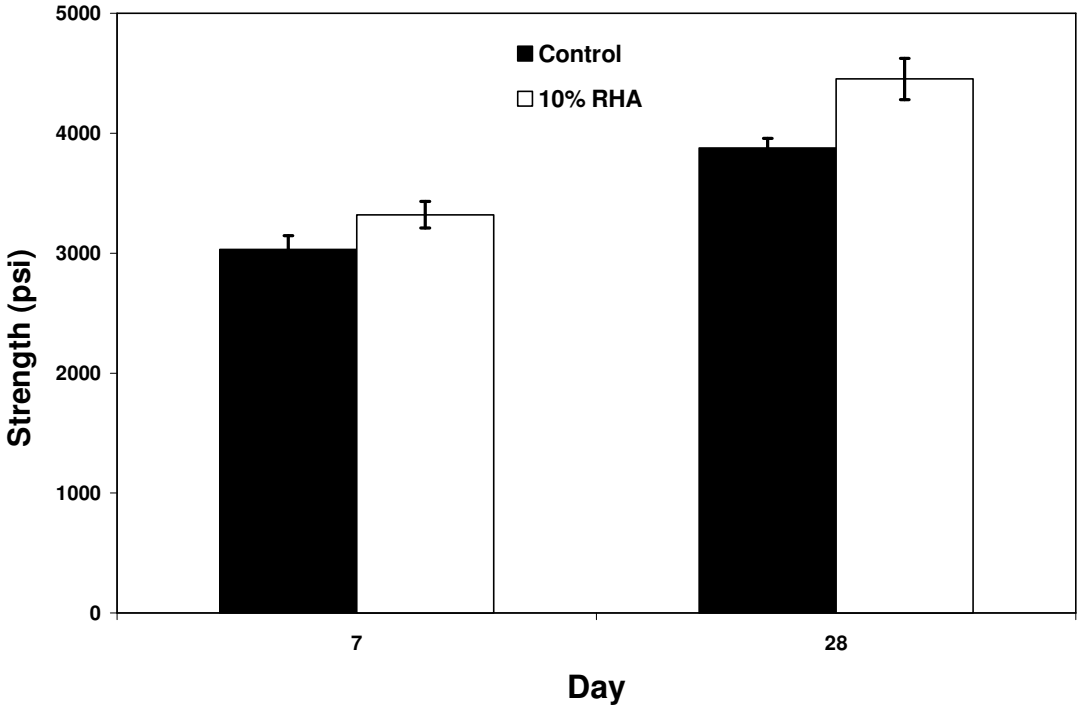

Figure 10.3: Strength results from concrete made with either 0% or 10% RHA

10.5 THERMOGRAVIMETRIC ANALYSIS

Another test done to determine the reactivity of the RHA, or its effectiveness in increasing the pozzolanic reaction was the thermogravimetric analysis of pastes made with Portland cement and varying contents of RHA. It is known that calcium hydroxide decomposes around 450℃, so the use of thermogravimetric analysis can measure the amounts of calcium hydroxide through the mass loss around 450℃. Paste samples were made and then pulverized to create a powder sample that was then used in the thermogravimetric analysis apparatus. The replacement levels of Portland cement with RHA were 0, 5, 10, 15, and 20%. The following reaction is associated with the decomposition of calcium hydroxide:

$$Ca(OH)_2 \rightarrow CaO + H_2O$$

By taking the original sample size, the percentage mass loss at 450℃, and the preceding reaction, the relative amounts of calcium hydroxide can be determined. An example of the calcium hydroxide decomposition using thermogravimetric analysis is shown in Figure 10.4. The samples were tested at 10 and 28 days to determine the effect of age on the relative amounts of calcium hydroxide. It was found that with the increasing amounts of RHA the amount of calcium hydroxide decreased. This is an indication that a pozzolanic reaction is occurring, and, therefore, the decrease in calcium hydroxide is associated with its conversion into C-S-H gel. The reactive silica found in the RHA is, therefore, reacting with the calcium hydroxide to form this added C-S-H gel.

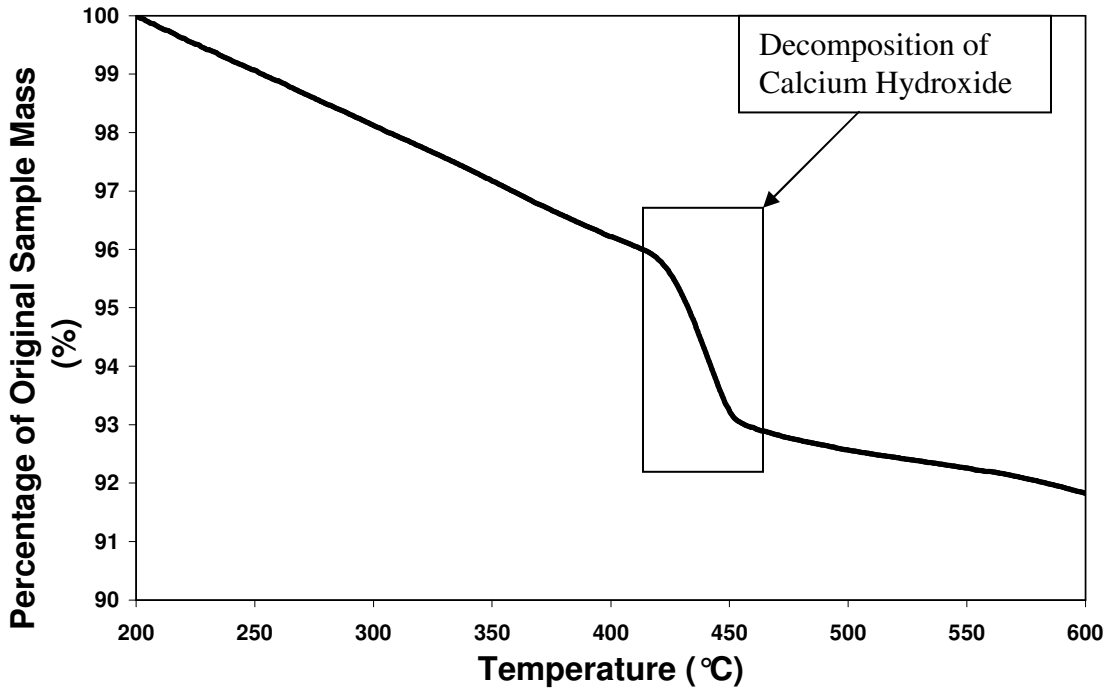

Figure 10.4: Example of Decomposition of Calcium Hydroxide using Thermogravimetric Analysis

The two following figures (Figure 10.5 and 10.6) show the relative amounts of calcium hydroxide with regard to the RHA replacement. The first graph is of the results after 10 days and the second graph is the results after 28 days. The graphs clearly show that with the increasing levels of RHA, the corresponding value of calcium hydroxide decreases. The values between 10 and 28 day vary slightly with the lower values being associated with the 28 day samples. This lower value of calcium hydroxide is associated with the on going curing of the sample.

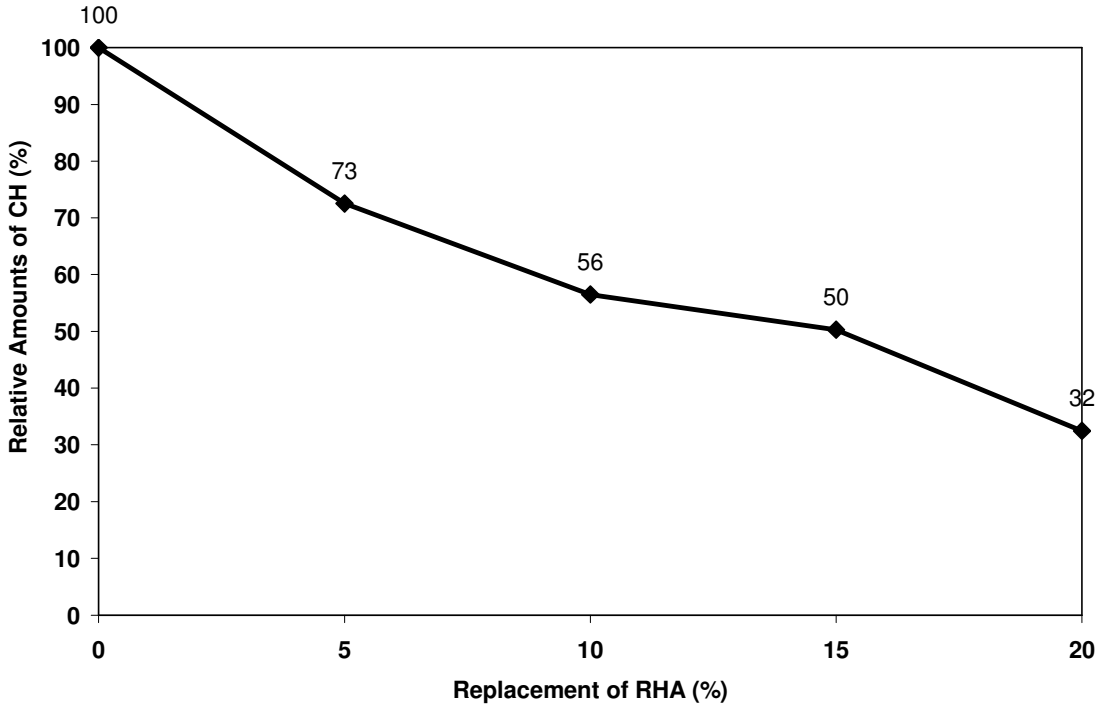

Figure 10.5: Relative amounts of Calcium Hydroxide vs. Replacement level of RHA for 10-day samples

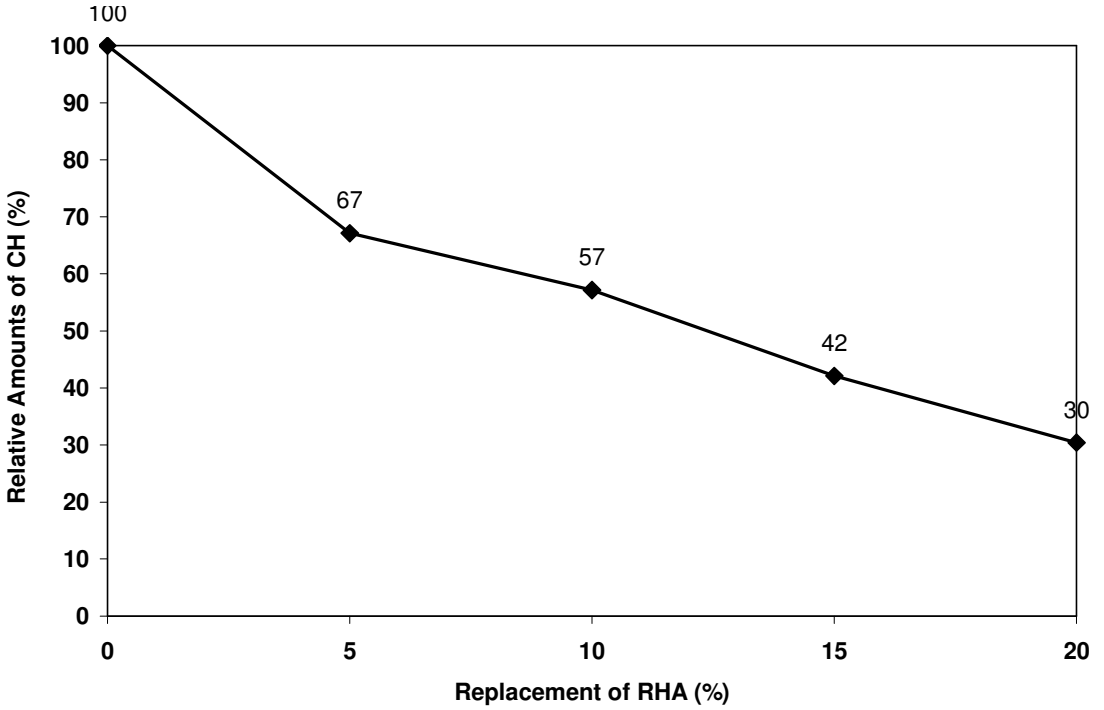

Figure 10.6: Relative amounts of Calcium Hydroxide vs. Replacement level of RHA for 28-day samples

10.6 SEM ANALYSIS OF RHA GRAINS

A final analysis of the physical properties of the RHA was done when the RHA alone was analyzed using a SEM. There were two ways that samples were prepared for analysis using the SEM. The two ways that samples were prepared were epoxy-embedded samples and powdered samples. The RHA was found to have a very large size distribution with irregularly shaped particles. The particles were found to very vesicular, which explains the high surface area found by the ChK Group. Figures 10.7 and 10.8 were taken from the epoxy embedded samples and show an individual RHA grain and a distribution of RHA grains, respectively. Figures 10.9 and 10.10 were taken from the powder samples and show an individual RHA grain and a distribution of RHA grains, respectively. These particles, being very vesicular, led to the early conclusion that the RHA may lead to mortar and concrete incorporating RHA being more permeable.

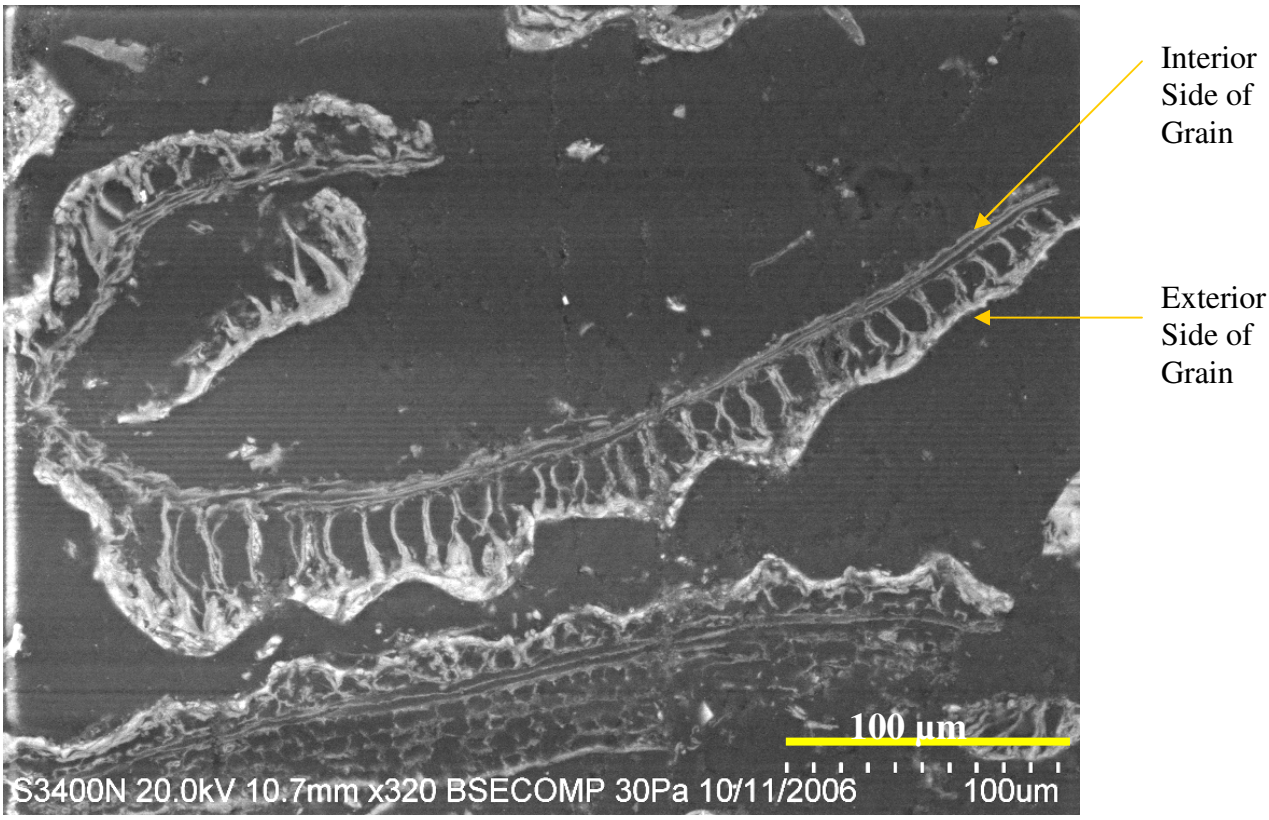

Figure 10.7: RHA grain taken from an epoxy-embedded sample

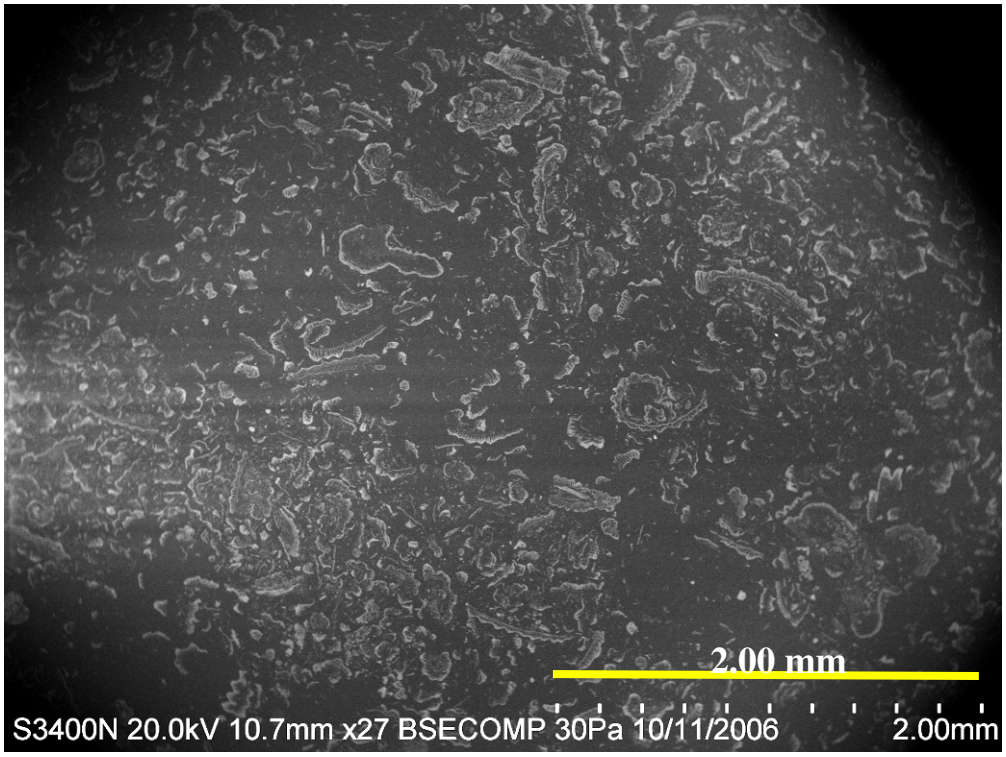

Figure 10.8: Distribution of RHA grains taken from an epoxy-embedded sample

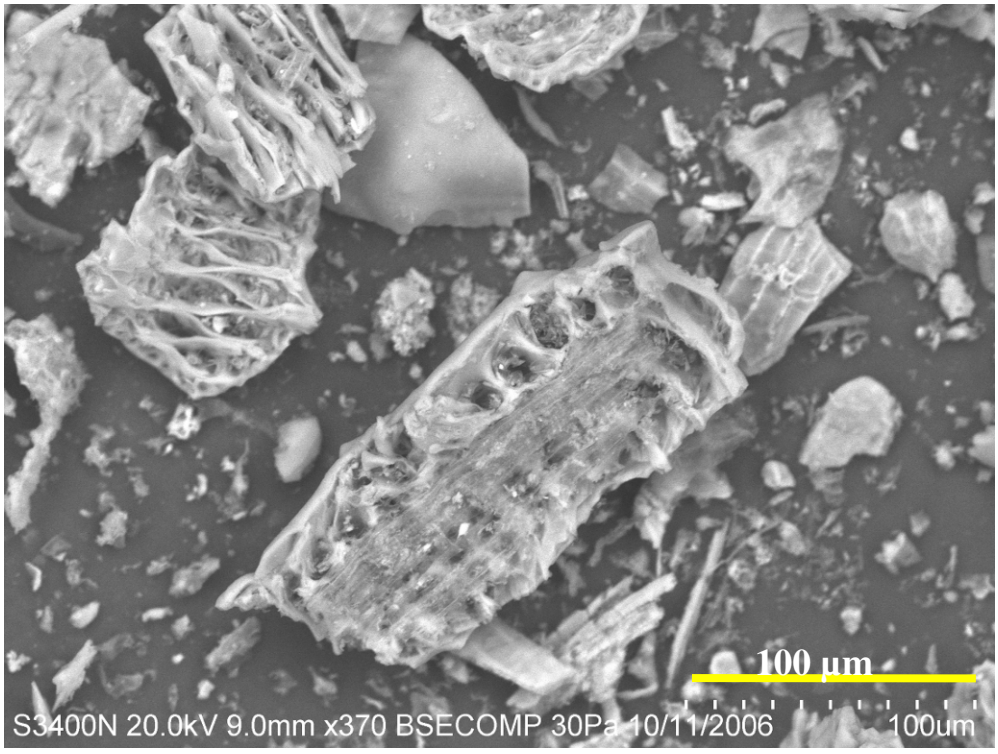

Figure 10.9: RHA grain taken from a Powder sample

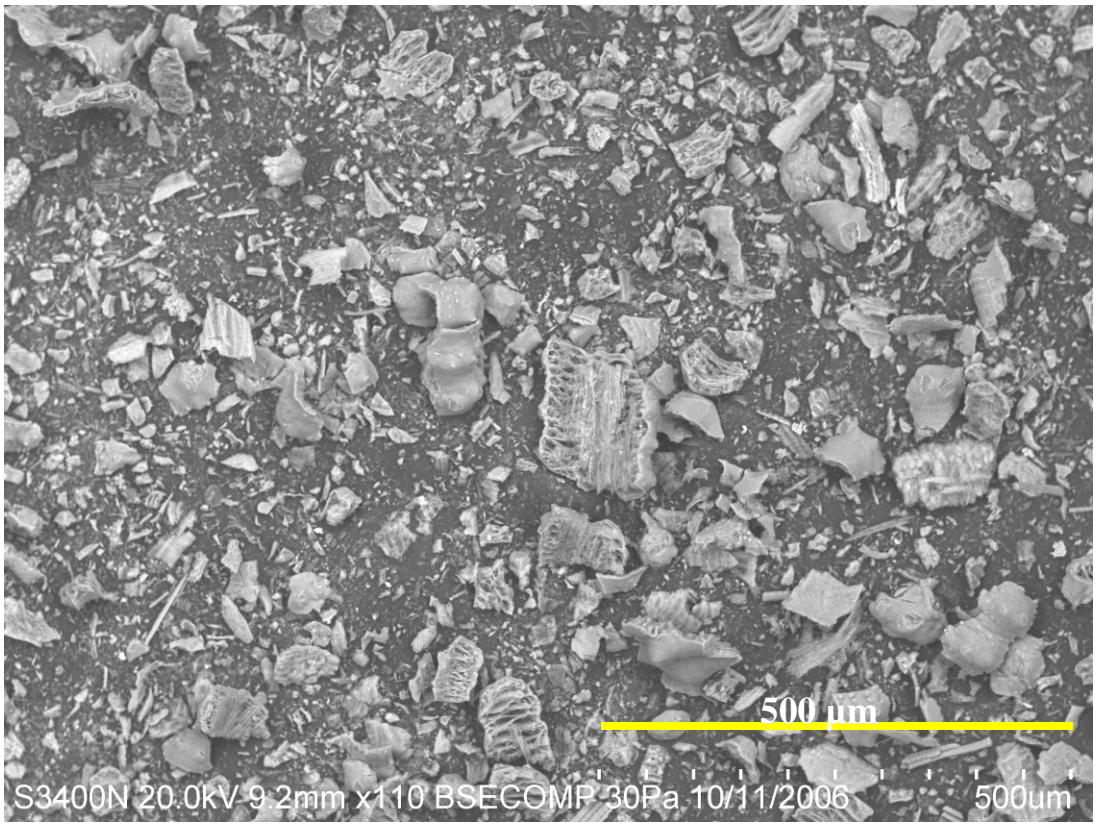

Figure 10.10: Distribution of RHA grains taken from a Powder sample

10.7 AFFECT OF INCORPORATING RHA ON PERMEABILITY

To determine the effect the RHA grains have on permeability in mortar and concrete, two tests were employed. These two tests were ASTM C 1202 (rapid chloride ion permeability) and ASTM C 642 (determination of absorption of concrete). These two tests were run in modification as well as in the standard form, with the only modification being the use of mortar instead of concrete. Permeability is a concern when the infiltration of external chemicals is of a concern, examples of this include the infiltration of salt water in oceanic environments or the infiltration of deicing chemicals used on runways or highways to remove ice. These chemical infiltrations can either lead to corrosion of reinforcing steel due to chloride or alkali-silica reaction problems due to the

infiltration of external alkalis. The vesicular nature of the RHA creates a concern, due to the potential to lead to increased permeability. Through the ASTM C 1202 test, it was seen that by increasing the amount of RHA, the rate at which charge was passed decreased. This leads to the conclusion that RHA does not increase permeability, but in fact decreases permeability with increased levels of RHA. The results of the modified test are presented in Figures 10.11 and 10.12, with Figure 10.11 being of the 14-day aged samples and Figure 10.12 being of the 28-day aged samples. The 10% RHA samples had higher variability than the others; therefore, only one sample is shown in Figures 10.11 and 10.12. The standard ASTM C 1202 results from the concrete made with RHA are displayed in Figure 11.13. This test was only done using a control concrete mixture and a 10% mass replacement of RHA with Portland cement, due to limited resources. The results again show that RHA helps to reduce permeability, because the control had a higher charge passing through the sample than did the 10% RHA sample. The results from the ASTM C 642 tests also indicated that decreased permeability occurs with increased RHA levels, but there was one irregularity; the 10% replacement level showed increased absorption in both the standard and modified ASTM C 642. It is unknown why this is occurring and is a recommendation for further investigation. The results from the absorption determination testing are found in Figure 10.14. From the results of these two tests, it was observed that permeability decreases with increased levels of RHA, with an exception occurring at 10% RHA. The ASTM C 1202 data was analyzed and the results of the findings can be found in Table 10.2.

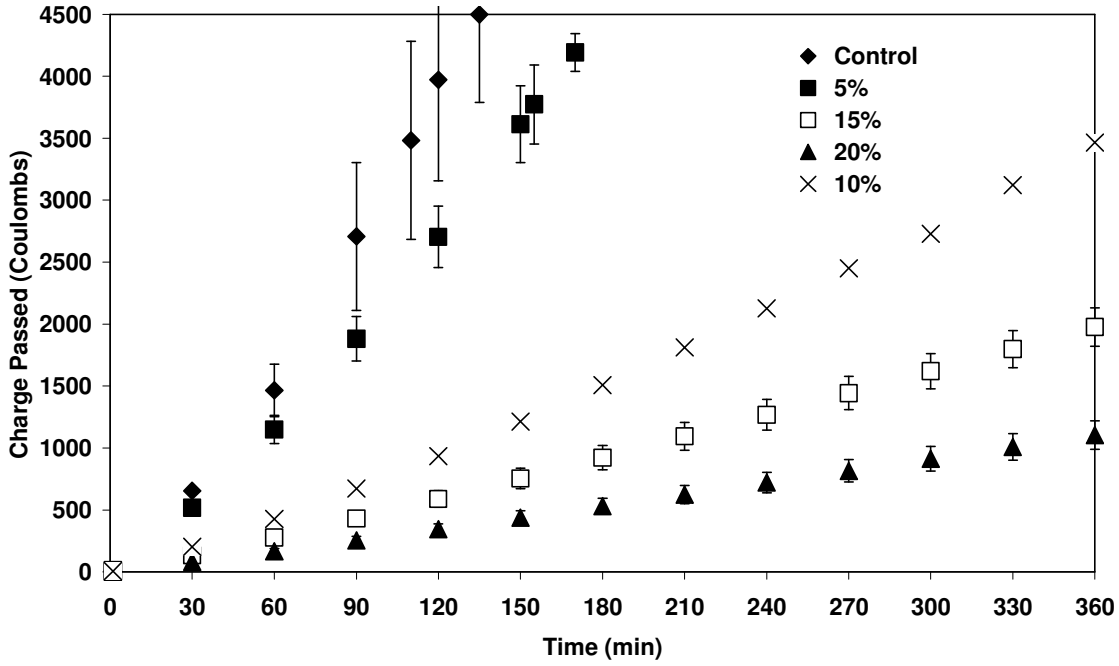

Figure 10.11: Modified ASTM C 1202 Results (14-Day)

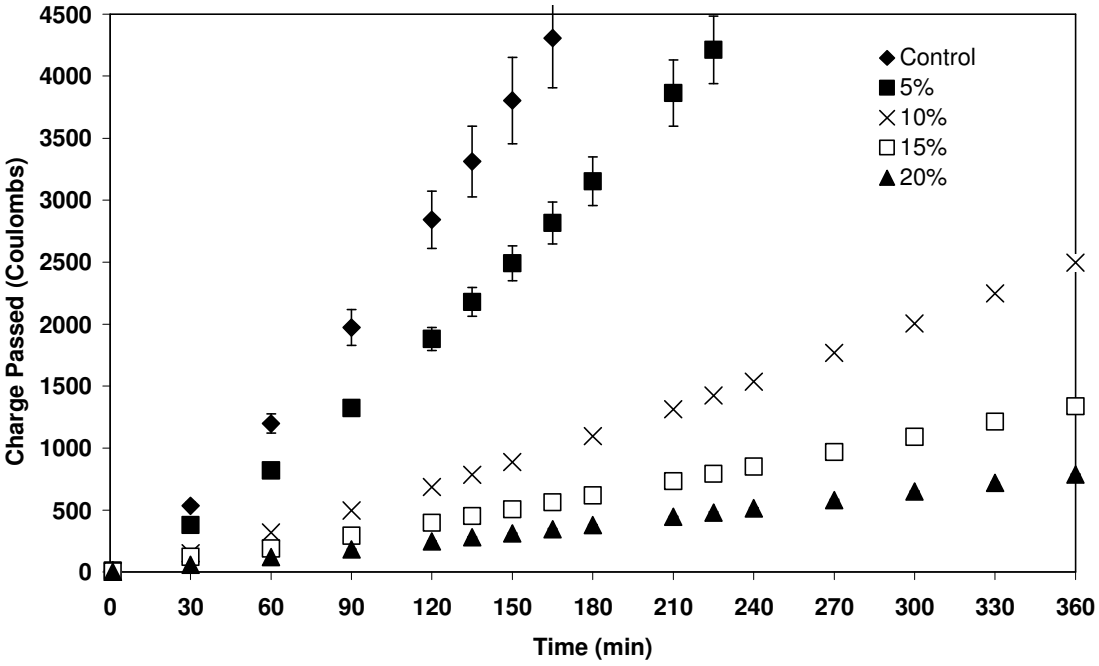

Figure 10.12: Modified ASTM C 1202 Results (28-Day)

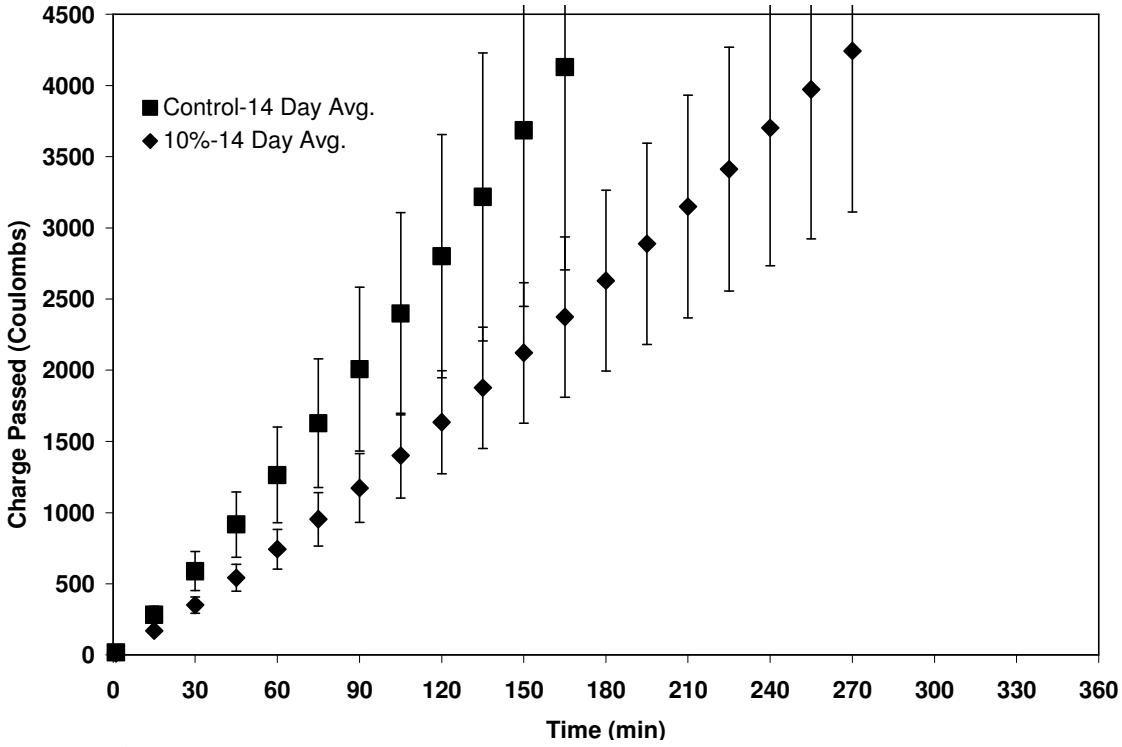

Figure 10.13: Standard ASTM C 1202 Results (Control and 10% RHA)

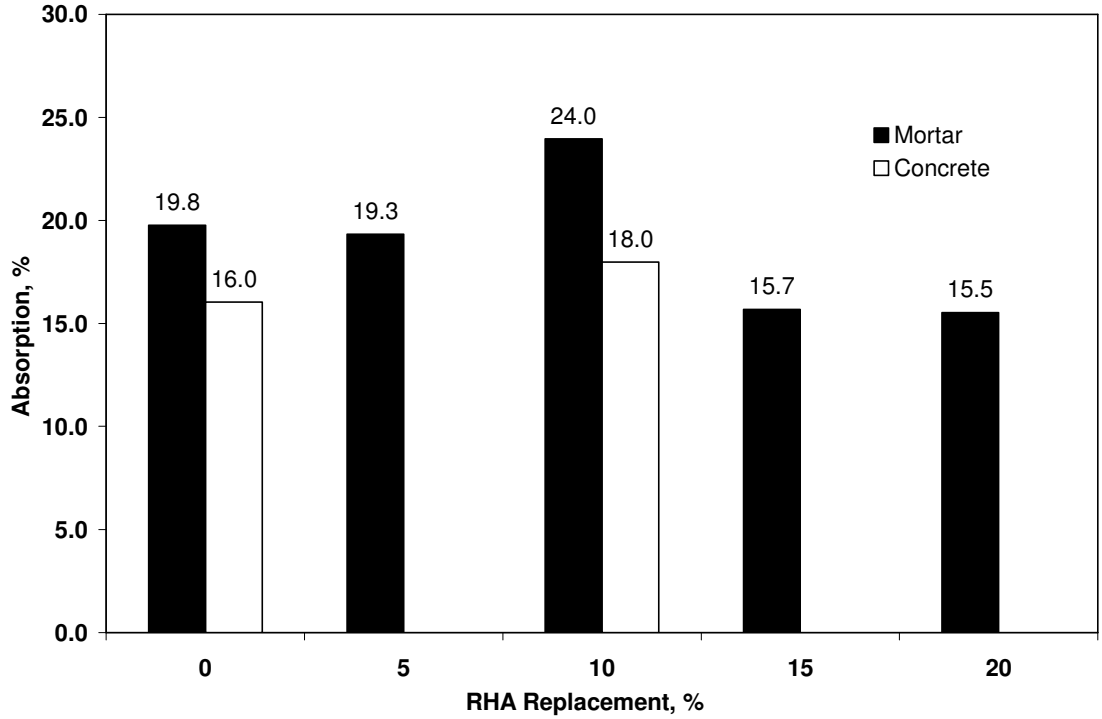

Figure 10.14: Standard and Modified ASTM C 642 Results

Table 10 .2: Statistical Analysis Results for the following tests:
ASTM C 1567, 1202, 311

RHA Dosage	0%	5%	10%	15%	20%
ASTM C 1567 14 Day Potassium 1N NaOH	A	A	B	C	-
ASTM C 1567 14 Day Potassium Acetate	A	A	B	C	-
ASTM C 1567 28 Day 1N NaOH	A	A	B	B	-
ASTM C 1567 28 Day Potassium Acetate	A	A	B	C	-
SAI No-Superplasticizer	A	A	B	C	D
SAI Superplasticizer	A	B	C	C	A
Modified ASTM C 1202 14-Day 120-Min	A	B	C	C	C
Modified ASTM C 1202 14-Day 120-Min	A	B	C	C/D	D

10.8 POTENTIAL ASR MITIGATION MEASURE

One of the main focuses of this study was to determine the mitigation potential of alkali-silica reaction through the use of RHA as a supplementary cementitious material. Through the literature review, it was found that, historically, the use of RHA aides in the mitigation of alkali-silica reaction. It was found that as the percentage of RHA replacement increases the expansions measured according to ASTM C 1567 decreased. From the data taken from this study's ASTM C 1567 test, it was found that the opposite occurs. This test was run twice due the discrepancy between the available literature's findings and this study's result. The results of the ASTM C 1567 test show that as the percentage of RHA increases so does the average expansion. The test was done in a standard form and also a modified form, with the modification being the replacement of the 1N NaOH solution with a potassium acetate solution. The average expansions are displayed for each of the two tests, for each of the solutions in Figures 10.15-10.22.

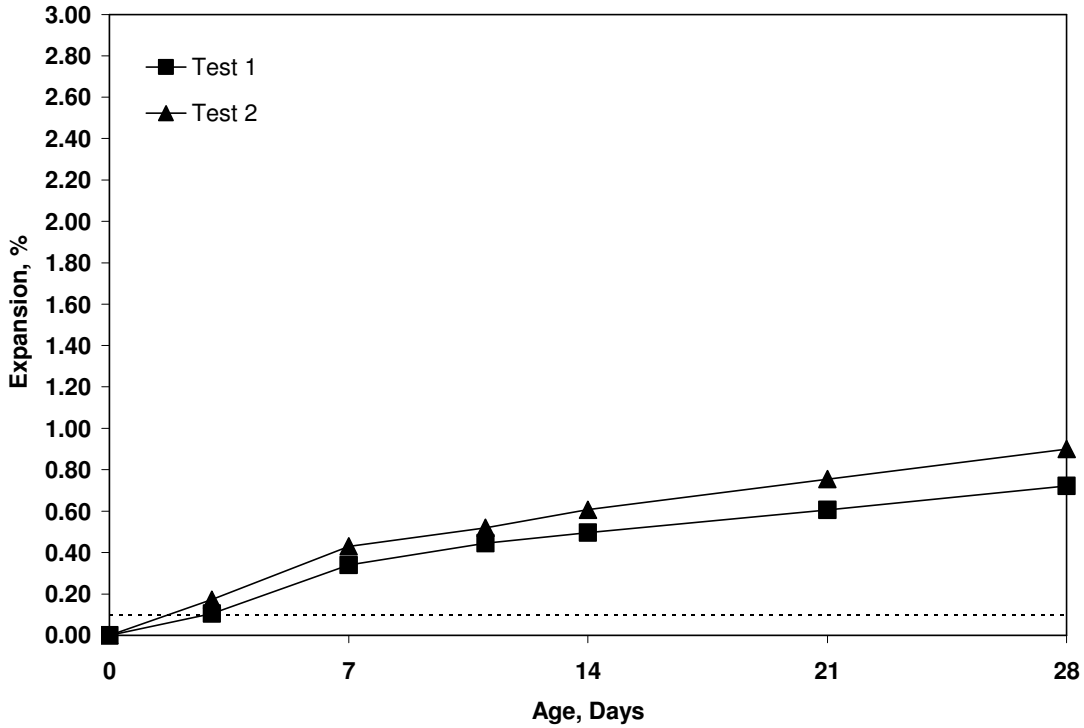

Figure 10.15: Control ASTM C 1567 Results from two tests for Potassium Acetate

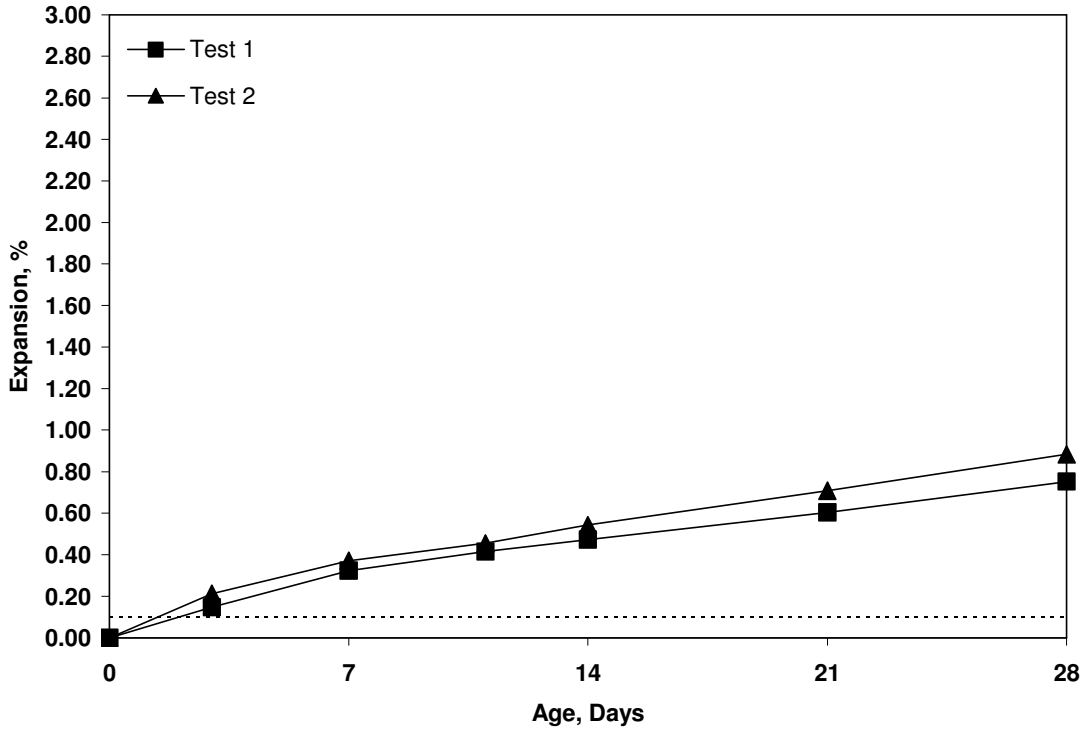

Figure 10.16: Control ASTM C 1567 Results from two tests for 1N NaOH

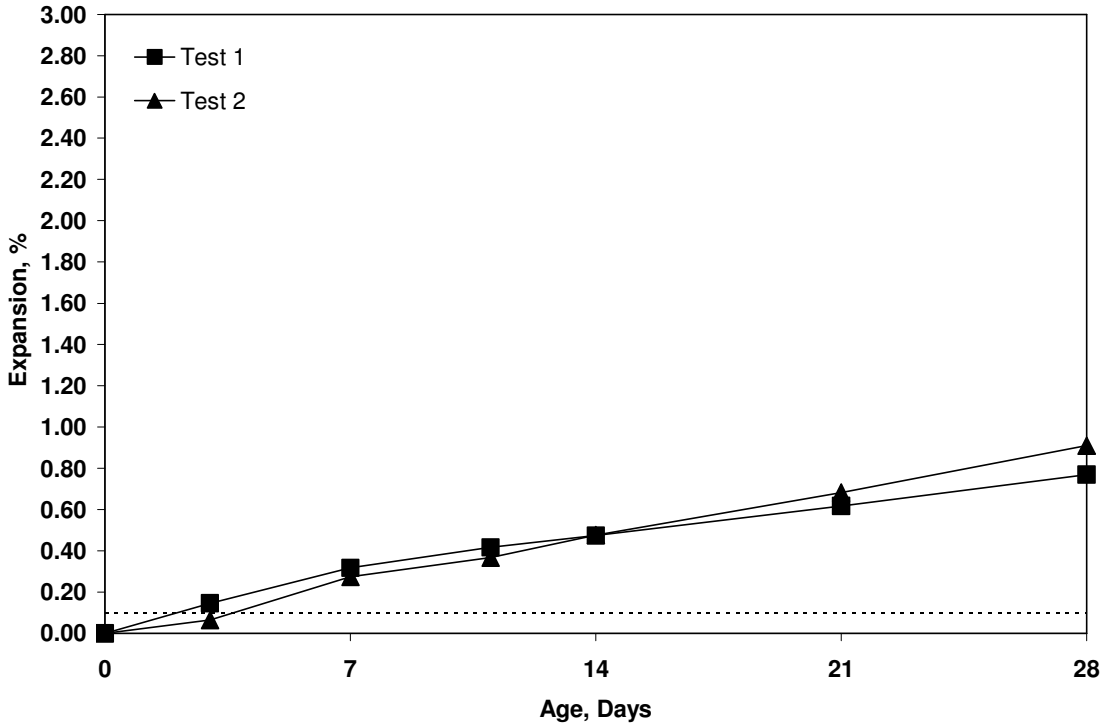

Figure 10.17: 5% RHA ASTM C 1567 Results from two tests for 1N NaOH

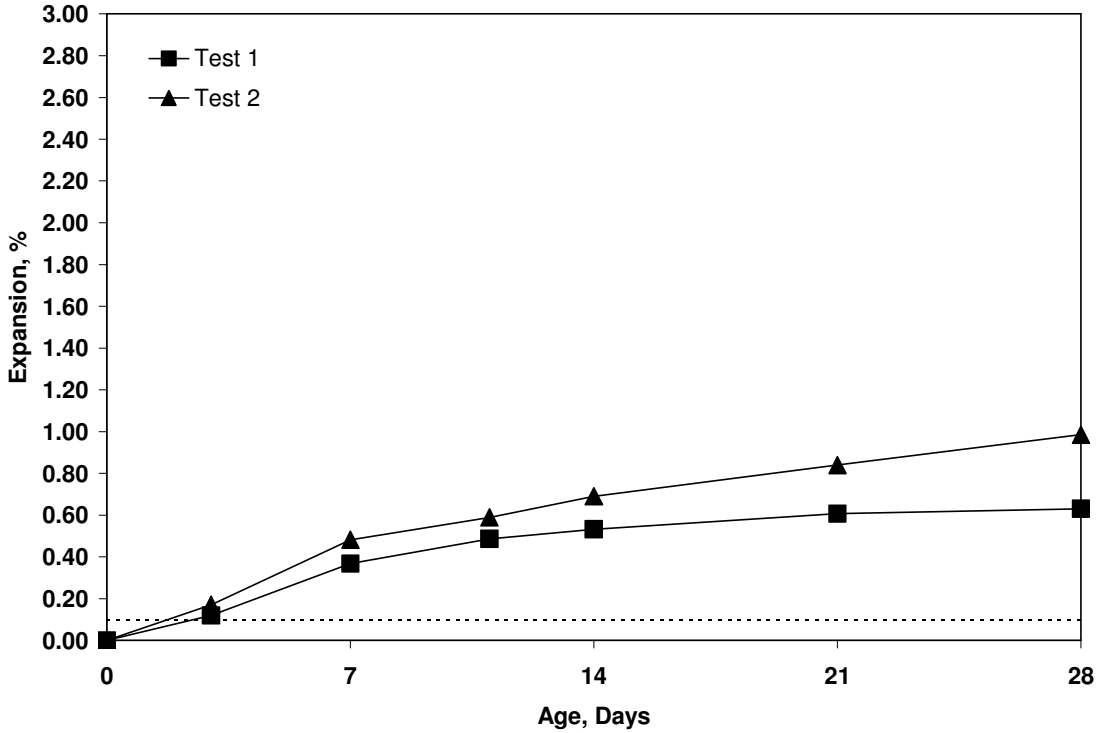

Figure 10.18: 5% RHA ASTM C 1567 Results from two tests for Potassium Acetate

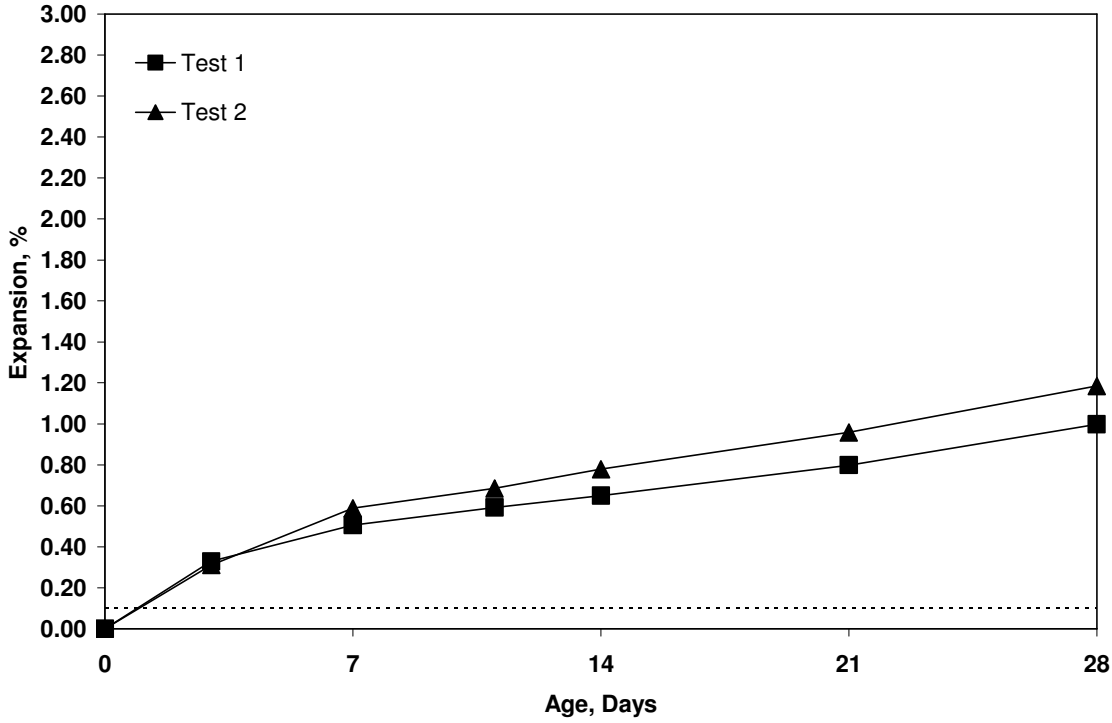

Figure 10.19: 10% RHA ASTM C 1567 Results from two tests for 1N NaOH

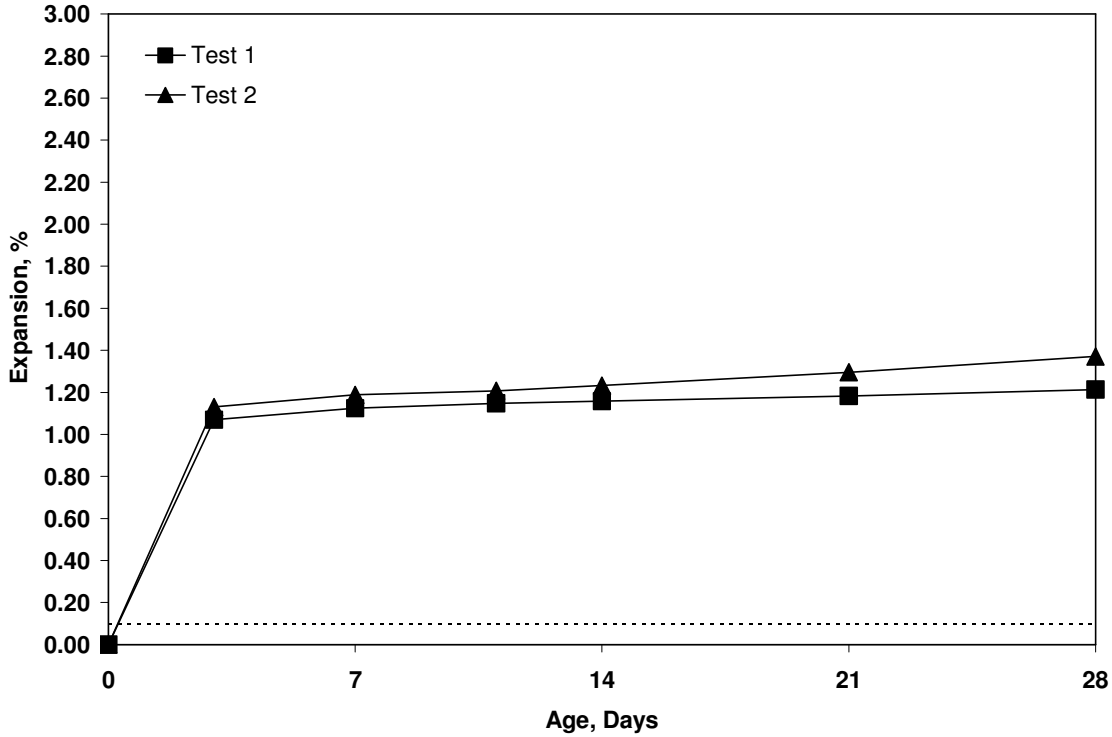

Figure 10.20: 10% RHA ASTM C 1567 Results from two tests for Potassium Acetate

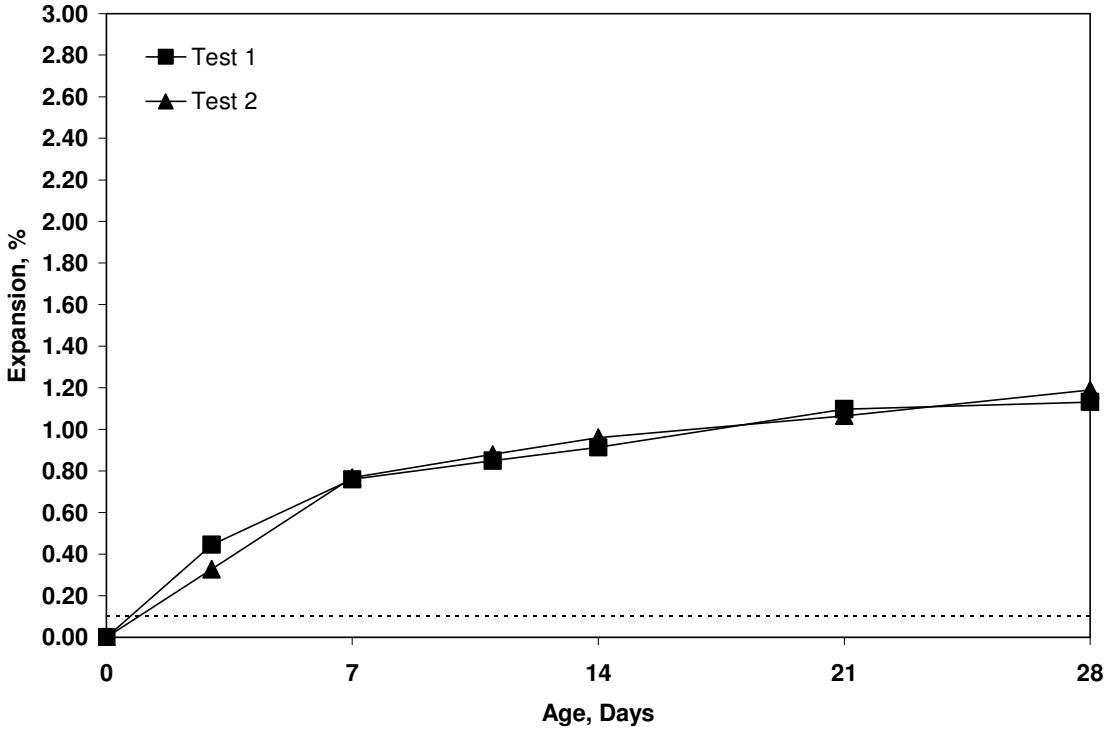

Figure 10.21: 15% RHA ASTM C 1567 Results from two tests for 1N NaOH

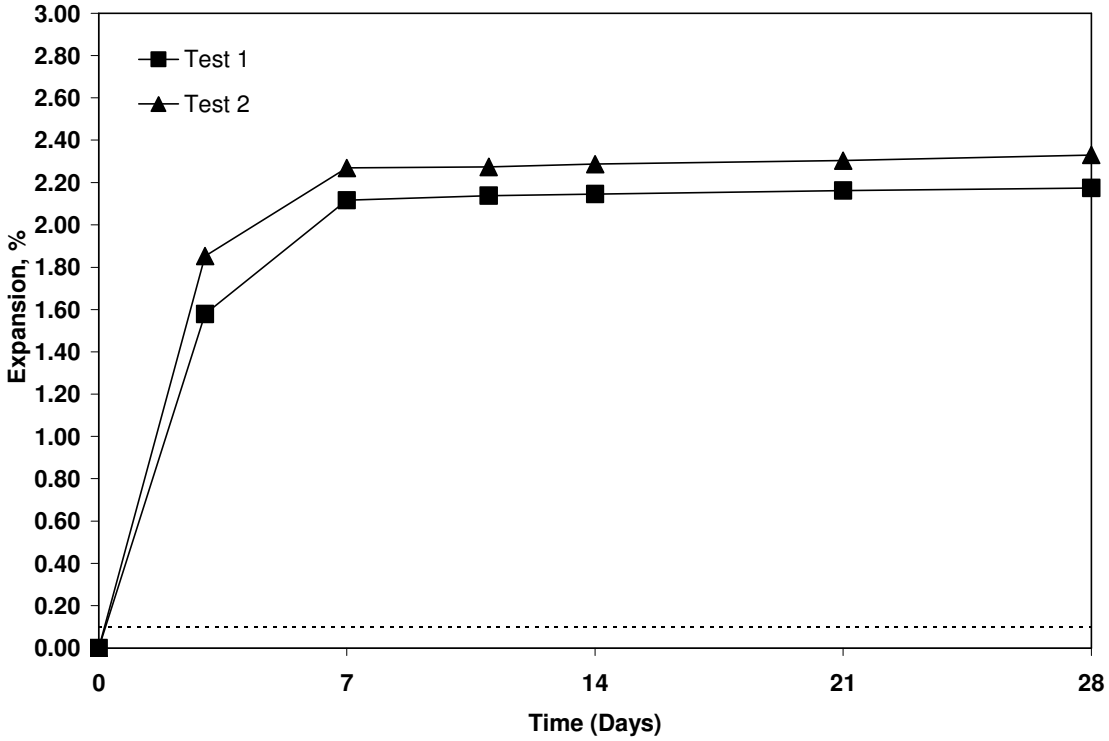

Figure 10.22: 15% RHA ASTM C 1567 Results from two tests for Potassium Acetate

The test was run with 20% RHA both times, but both tests resulted in the mortar bars deteriorating due to excessive expansions. The coefficient of variation of each test was computed and compared to the 2.94% limit listed in the standard. The tests met the requirement except for the 15% replacement, which it is hypothesized that this is due to 15% having such large expansions. The tests were also compared using an analysis of variance table using a confidence interval of 95%, or also written as an α value of 5%. The results of the analysis of variance table showed that for all the tests except for 15% the data was significantly different between the two tests, but the overall trend lines of the data are all similar for each test for each replacement level. There is a difference in results expected because the two tests were from different batches and were prepared at different times. The overall averages of the two tests for each solution are displayed in Figures 10.23 and 10.24.

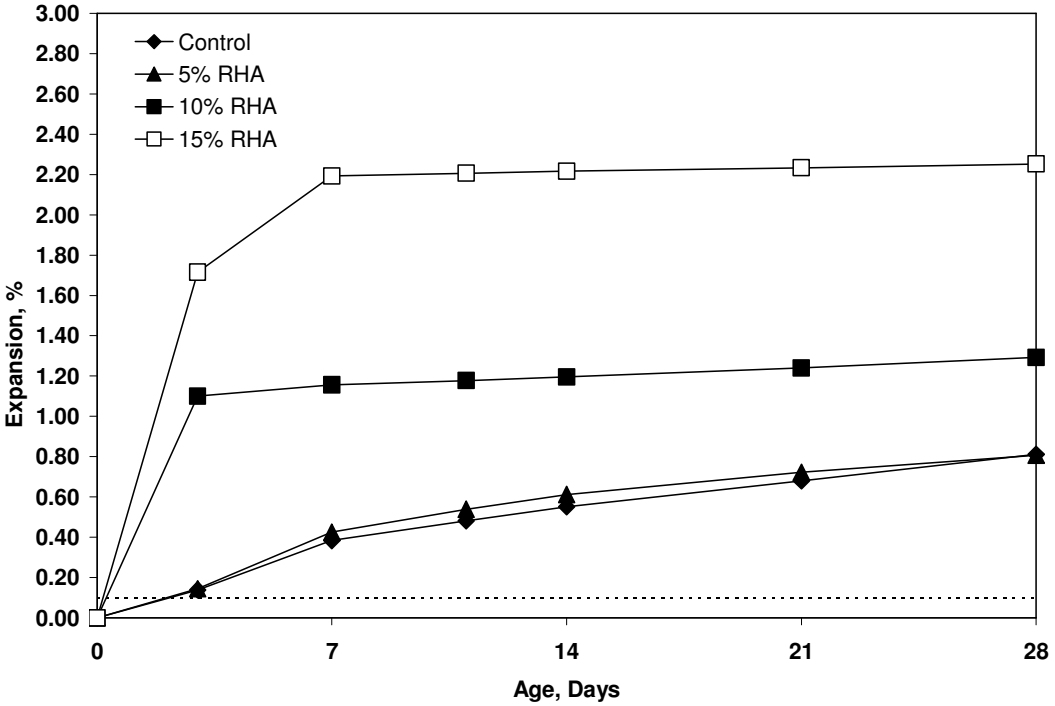

Figure 10.23: ASTM C 1567 Avg. Results from the two tests for each Potassium Acetate Solution

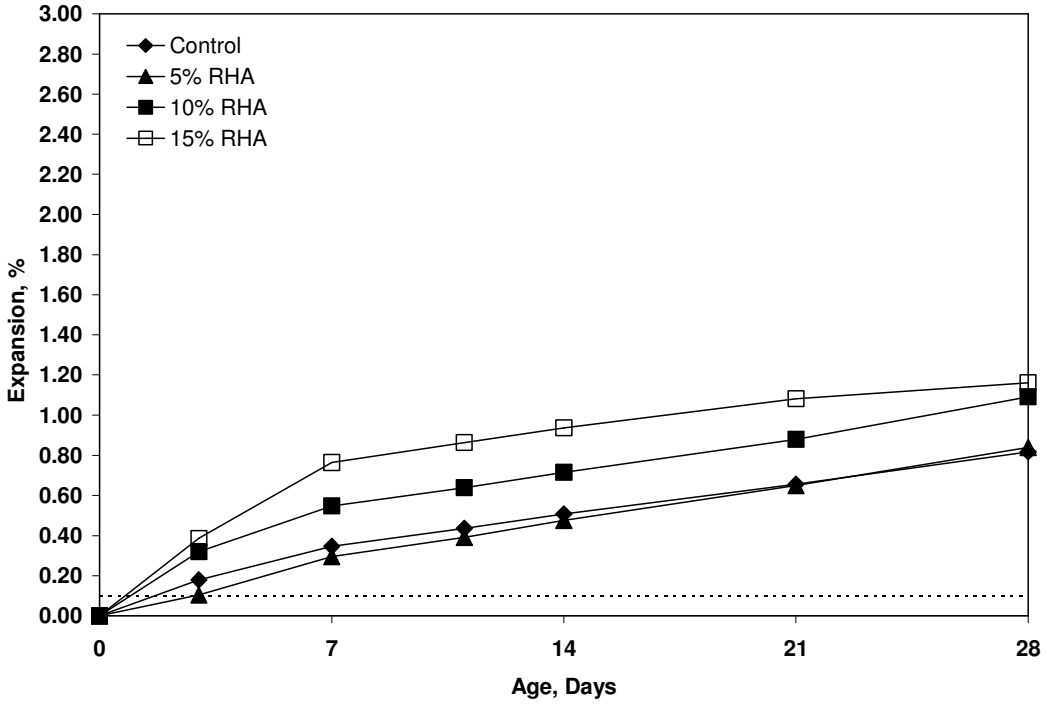

Figure 10.24: ASTM C 1567 Avg. Results from the two tests for each 1N NaOH Solution

For the lower percentages of RHA, 0 and 5%, the results between the two solutions were very similar and through an analysis of variance table it was determined that the values of expansion were not statistically different for both solutions. This indicates that with 5% RHA replacement, durability with regard to resistance to ASR is similar to that of the control. For the higher replacement levels (10 and 15%) it was determined that there results were statistically different when compared to the control and 5% RHA replacement and also between each other. This indicates that the expansions did increase as the replacement level increased past 5%, which indicates that with RHA replacement levels greater than 5%, RHA fails to be successful as an ASR mitigator. As for differences between the standard solution (1N NaOH) and the modified solution (potassium acetate), it was determined statistically that the results were different for all the replacement levels except the control. For all the cases where RHA was used, it was found that the potassium acetate solution produced higher expansion levels, which is expected due to the fact that more available alkalis are present in the potassium acetate solution. The results of this test are also shown in Table 10.2. Each replacement level is given a letter and replacement levels with the same letter indicate that there is not a significant difference between the two, and different letters, therefore, mean that the results were significantly different. These differences were determined using SAS, which is a statistical analysis computer program.

10.9 SEM INVESTIGATION OF MORTAR BAR SAMPLES

An investigation of the mortar bars on the microscopic level confirmed the results from the ASTM C 1567 tests, meaning that increased cracking was seen as the replacement level increased. This was the expected result, because with the mortar bars increased expansion due to ASR gel expanding there is an associated increase in tensile forces within the mortar matrix which then causes the matrix to deteriorate through cracking. The control samples were analyzed using the SEM, but due to data corruption, the files were inoperable. An example of the pictures and EDX spectra for an individual replacement level is displayed in Figure 10.25, with the rest of the visual data located in the appendix section K. These samples were taken after the completed ASTM C 1567 test, which lasted 28 days. The EDX attachment was calibrated before each test using a piece of copper tape.

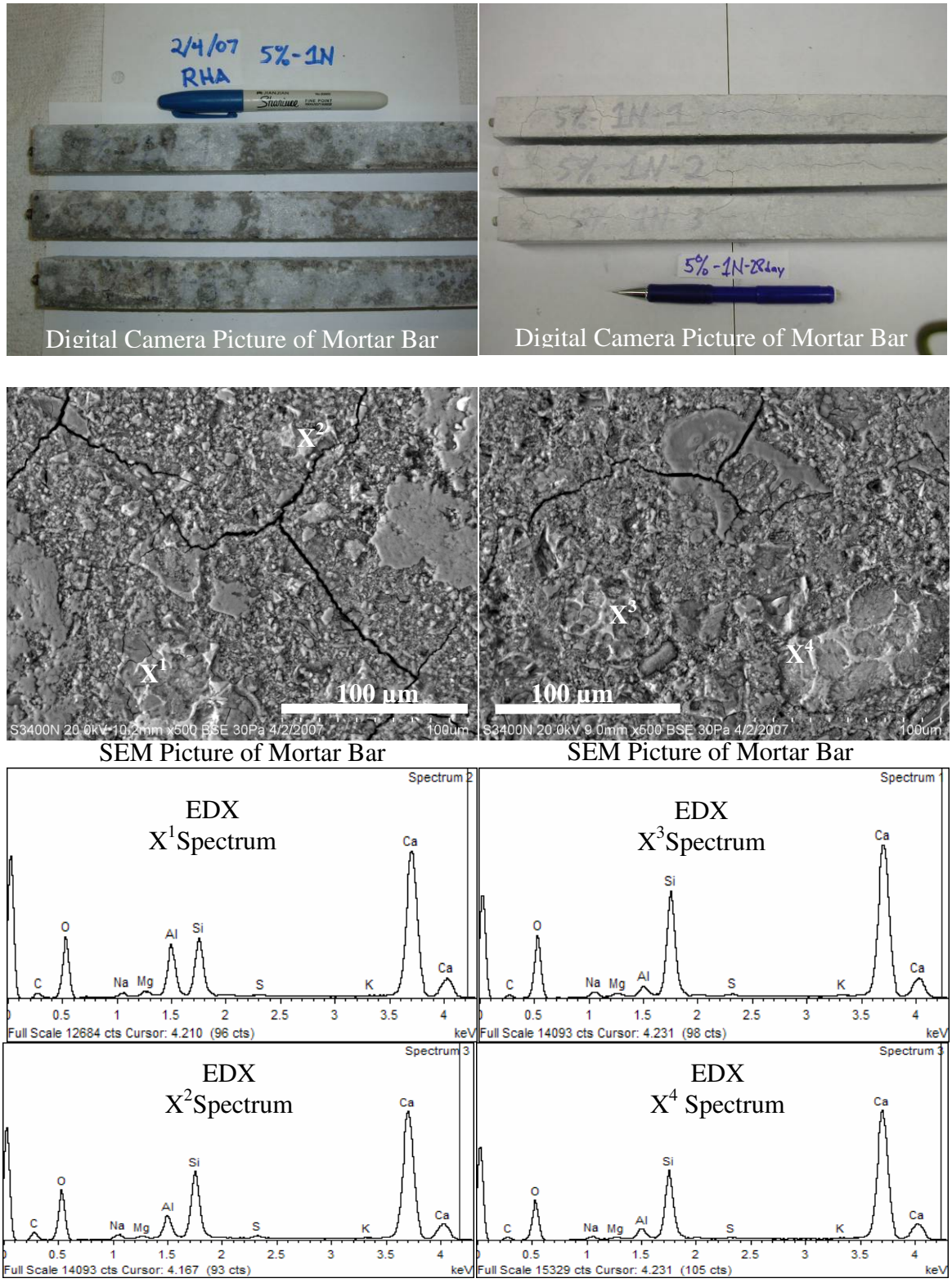

Figure 10.25: Digital Photographs with SEM and EDX Results for Mortar Bar Samples

11. CONCLUSIONS

- The new process to create RHA with lower carbon contents created by the ChK Group Inc. is successful in reducing carbon contents, due to the fact that x-ray fluorescence analysis showed that the RHA created by the new process had around 0.24% carbon compared to 3-18% for other RHAs.

- The high surface area of the RHA (35 m^2/g) increases its reactivity, but also can increase the water demand in mortar and concrete with RHA incorporated.

- The density of the RHA was found to be low (2.08) when compared to Portland cement.

- The Strength Activity Index (SAI or PAI) for the RHA showed that there was an optimum dosage between 10 and 15%, but it was also seen from the results of the two tests a superplasticizer is needed to overcome the increased water demand created by the incorporation of RHA. This increased strength gain is associated with the added pozzolanic reaction.

- When RHA is incorporated in concrete at a mass replacement rate of 10%, then strength is increased when compared to a control concrete at early ages and also later ages.

- Thermogravimetric analysis showed that there is a pozzolanic reaction occurring, due to the fact that as the dosage rate increases the amount of calcium hydroxide dropped, which indicates a strong pozzolanic reaction occurring to convert the calcium hydroxide to C-S-H gel.

- The individual RHA grains are very vesicular, which was confirmed by SEM analysis, explaining the high surface area and increased water demand caused by the incorporation of RHA.

- The permeability of mortars and concrete made with RHA decreases, which is helpful to create more durable materials. This fact was proven by standard and modified ASTM C 1202 and C 642 tests.

- RHA, in its present form, was not successful in mitigating ASR, which evident after the two ASTM C 1567 tests showed increased expansions with increased dosage levels. The 5% dosage behaved the same as the control and was concluded to be not significantly different statistically, where the other dosage levels were determined to be significantly different. The potassium acetate solution caused increased expansions when compared to the standard 1N NaOH solution.

- SEM investigation of the mortar bar samples showed that as the dosage level increased so did the amount of micro cracking. This confirmed the statement that the incorporation of RHA, in its present form, was not successful in mitigating ASR

12. RECOMMENDATIONS

12.1 RECOMMENDATIONS FROM THE CONCLUSIONS

- Maximum strength gain was seen at a mass replacement level between 10 and 15%.

- A superplasticizer or high-range water reducer is needed to overcome the increased water demand caused by the very vesicular rice husk ash grain.

- With increasing rates of RHA replacement a decrease in permeability is seen, which is important when dealing with chemical ingression into the concrete or mortar creating a more durable material. Having said this it is recommended to use RHA when chemical ingression is a factor.

- A dosage rate of 5% RHA, in its present form, was equivalent to the control mortar with regard to ASR Mitigation, and increasing dosages increase the amounts of expansion, so use 5% RHA or less when concerned with ASR problems.

12.2 RECOMMENDATIONS FOR FURTHER RESEARCH

- An investigation into the effect of grinding time on the reactivity of the RHA and also the effect on mortar and concrete properties using the RHA created by the ChK Group Inc.

- A further investigation into the ASR mitigation potential using a more accurate test procedure such as ASTM C 1293 to confirm the results of the ASTM C 1567 test.

- An investigation into the differences created using a volumetric replacement instead of the mass replacement procedure that was used.

- A more in depth study into the effect of incorporating the RHA into Portland cement concrete, utilizing multiple replacement levels of RHA.

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APPENDICES

A. AGGREGATE SEM AND EDX INVESTIGATION

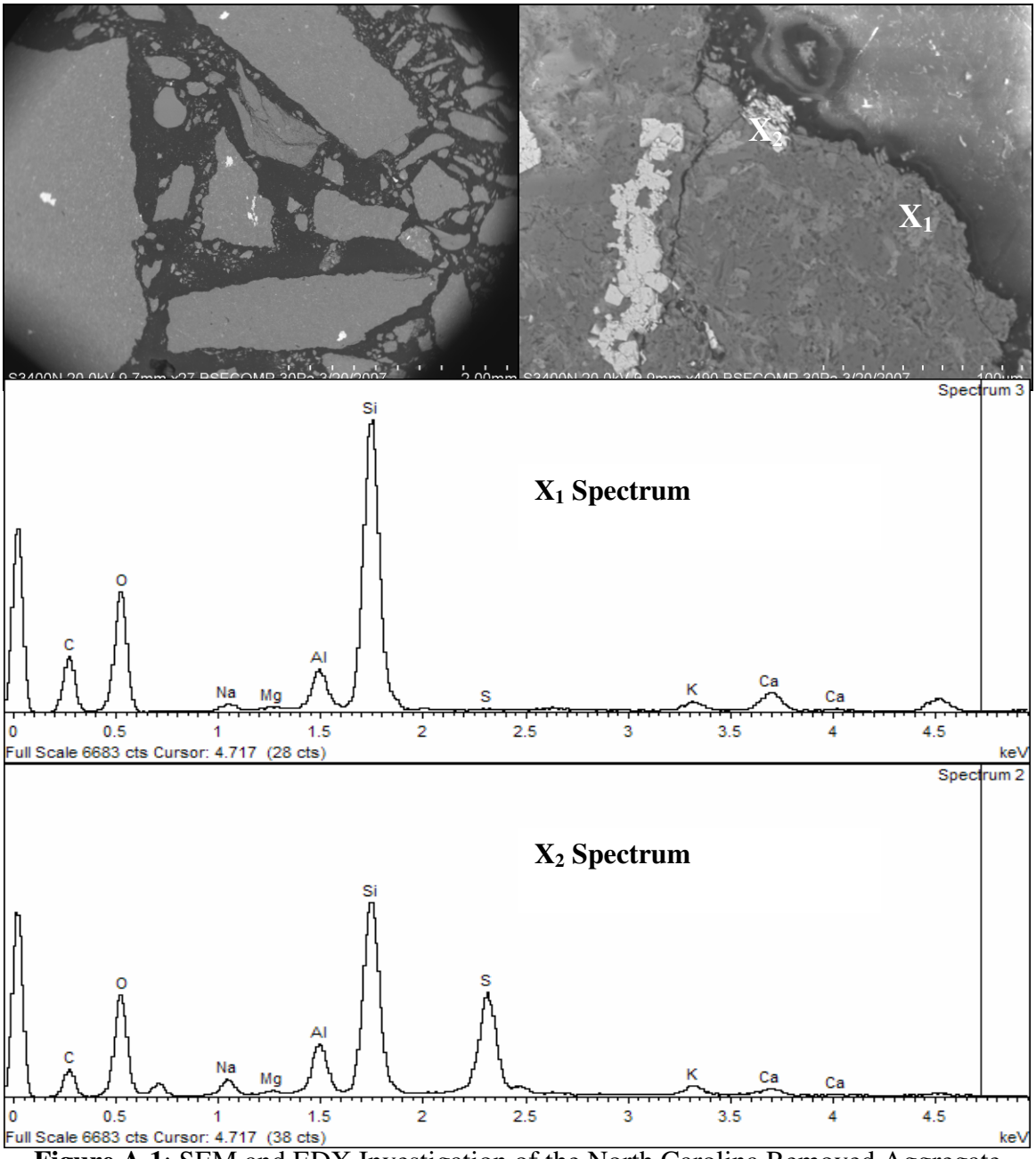

Figure A.1: SEM and EDX Investigation of the North Carolina Removed Aggregate

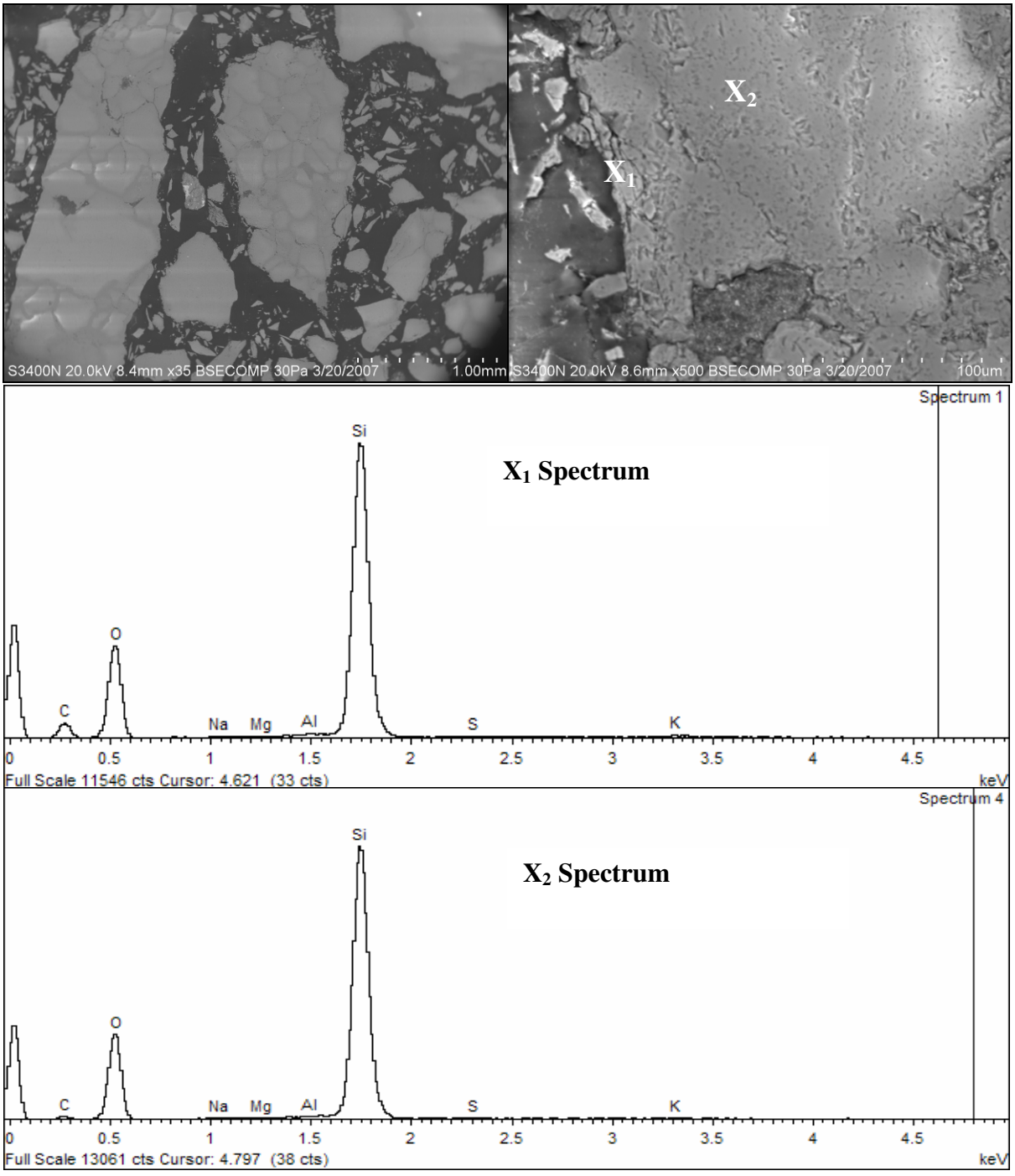

Figure A.2: SEM and EDX Investigation of the South Dakota Removed Aggregate

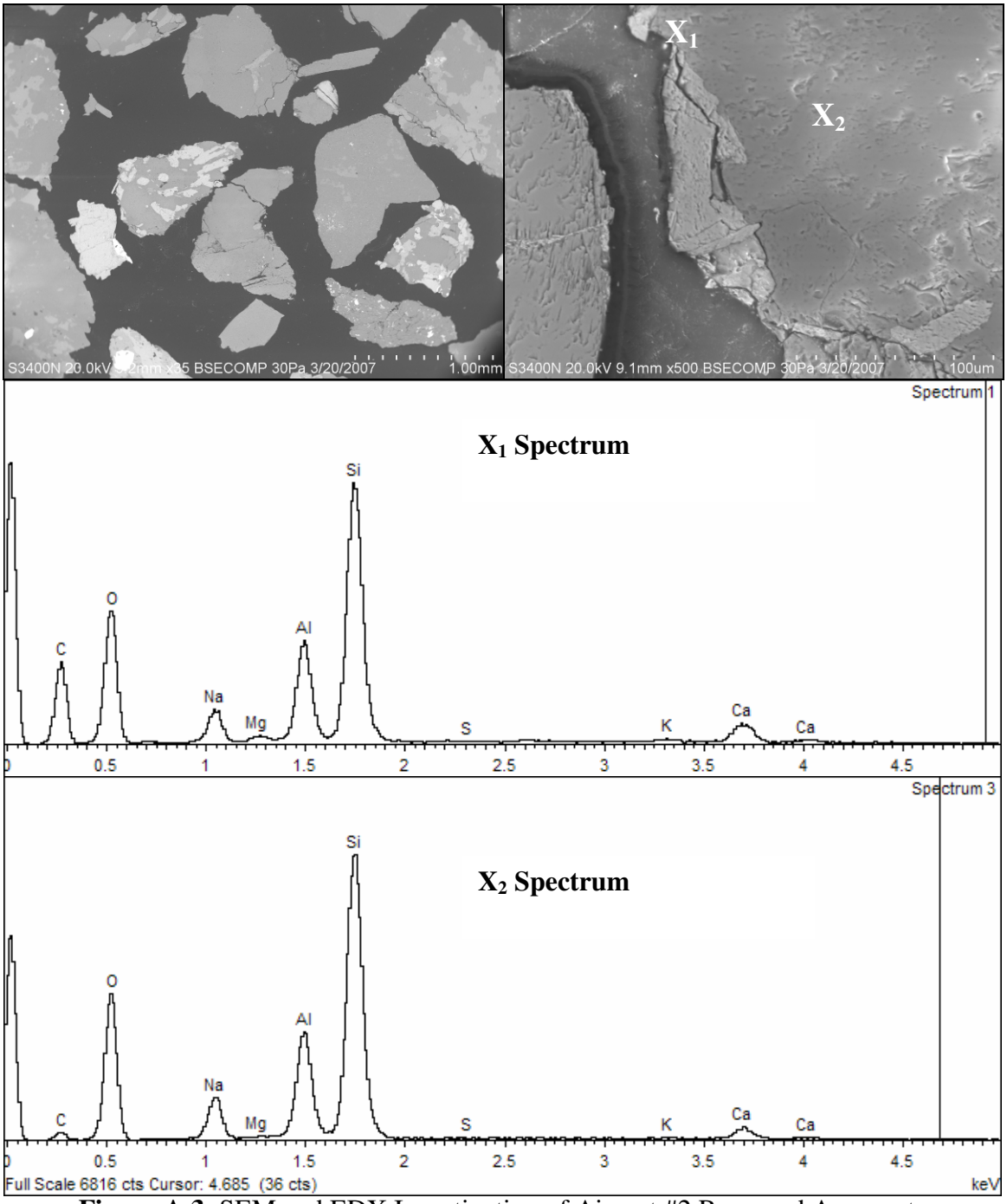

Figure A.3: SEM and EDX Investigation of Airport #2 Removed Aggregate

B. AGGREGATE REMOVAL PROCESS

Figure B.1: Visual Progression of Aggregate Removal for New Mexico Samples in 1N NaOH

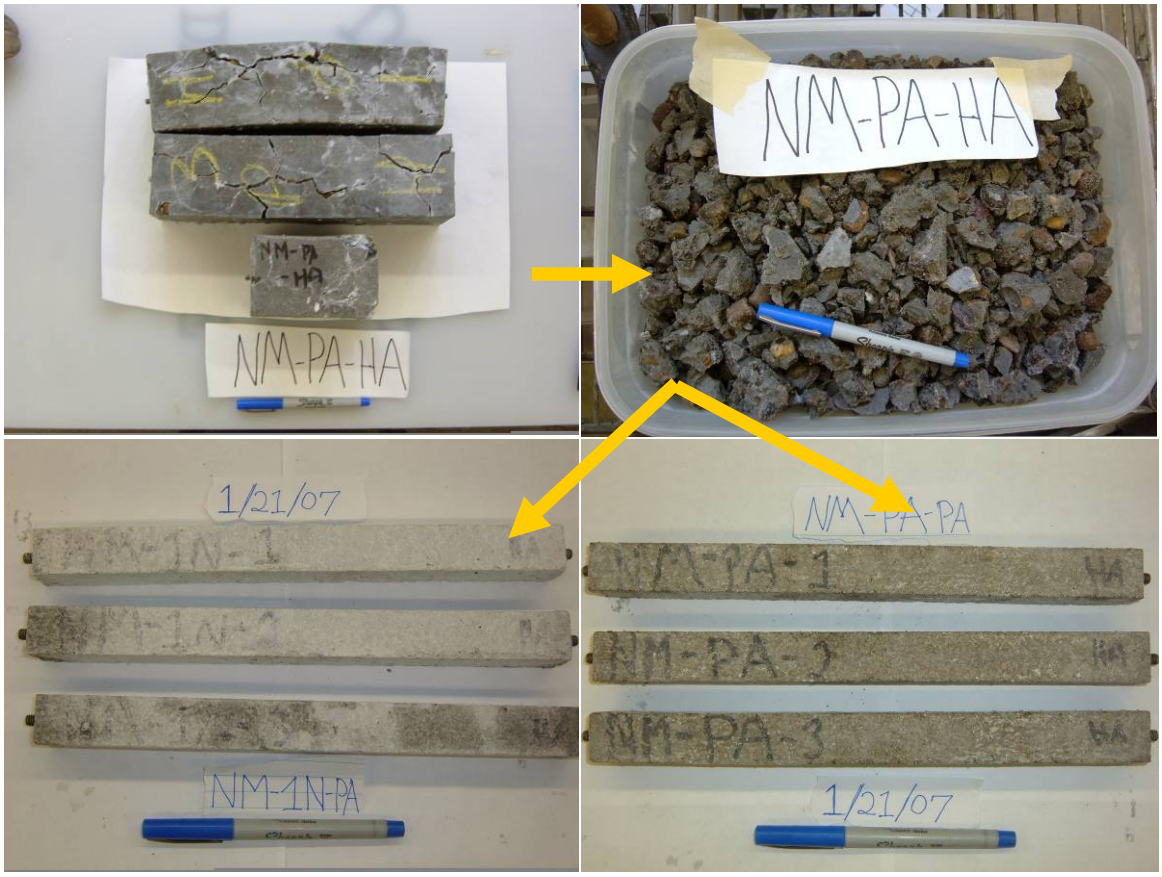

Figure B.2: Visual Progression of Aggregate Removal for New Mexico Samples in Potassium Acetate

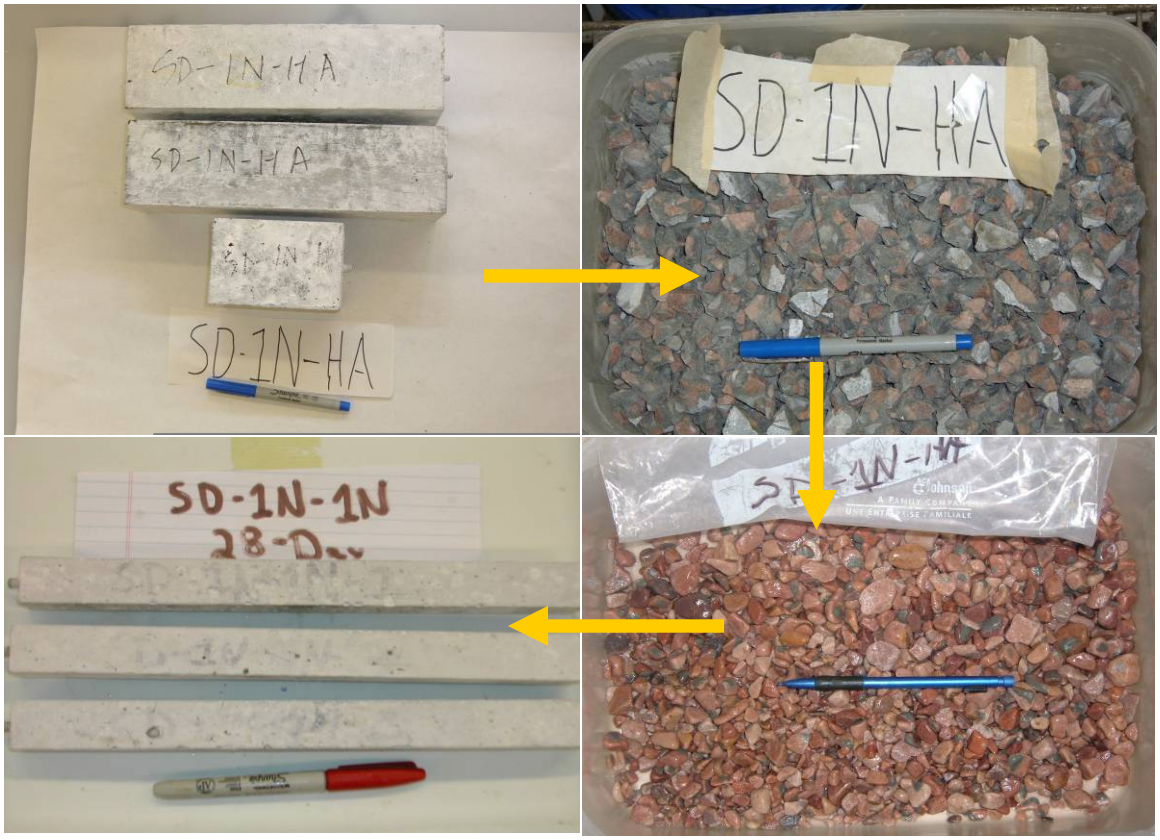

Figure B.3: Visual Progression of Aggregate Removal for South Dakota Samples in 1N Sodium Hydroxide

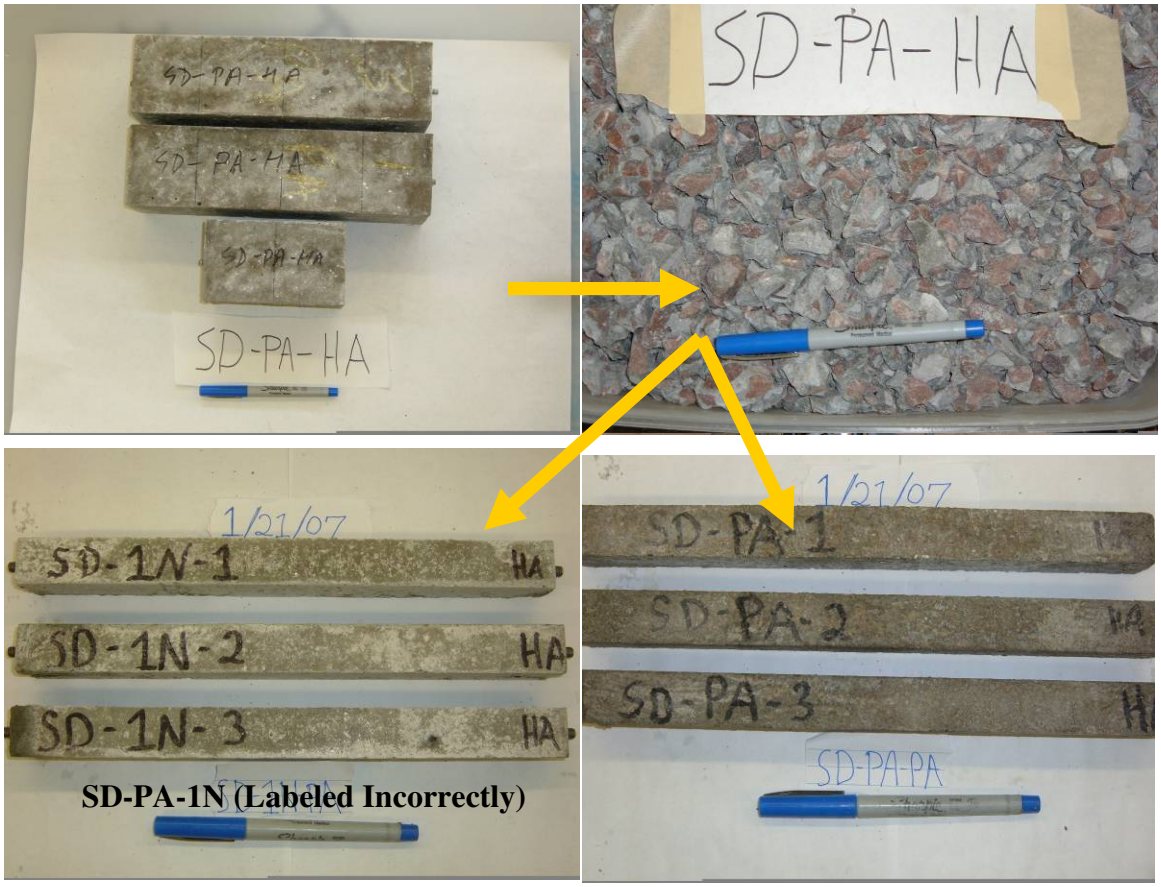

Figure B.4: Visual Progression of Aggregate Removal for South Dakota Samples in Potassium Acetate

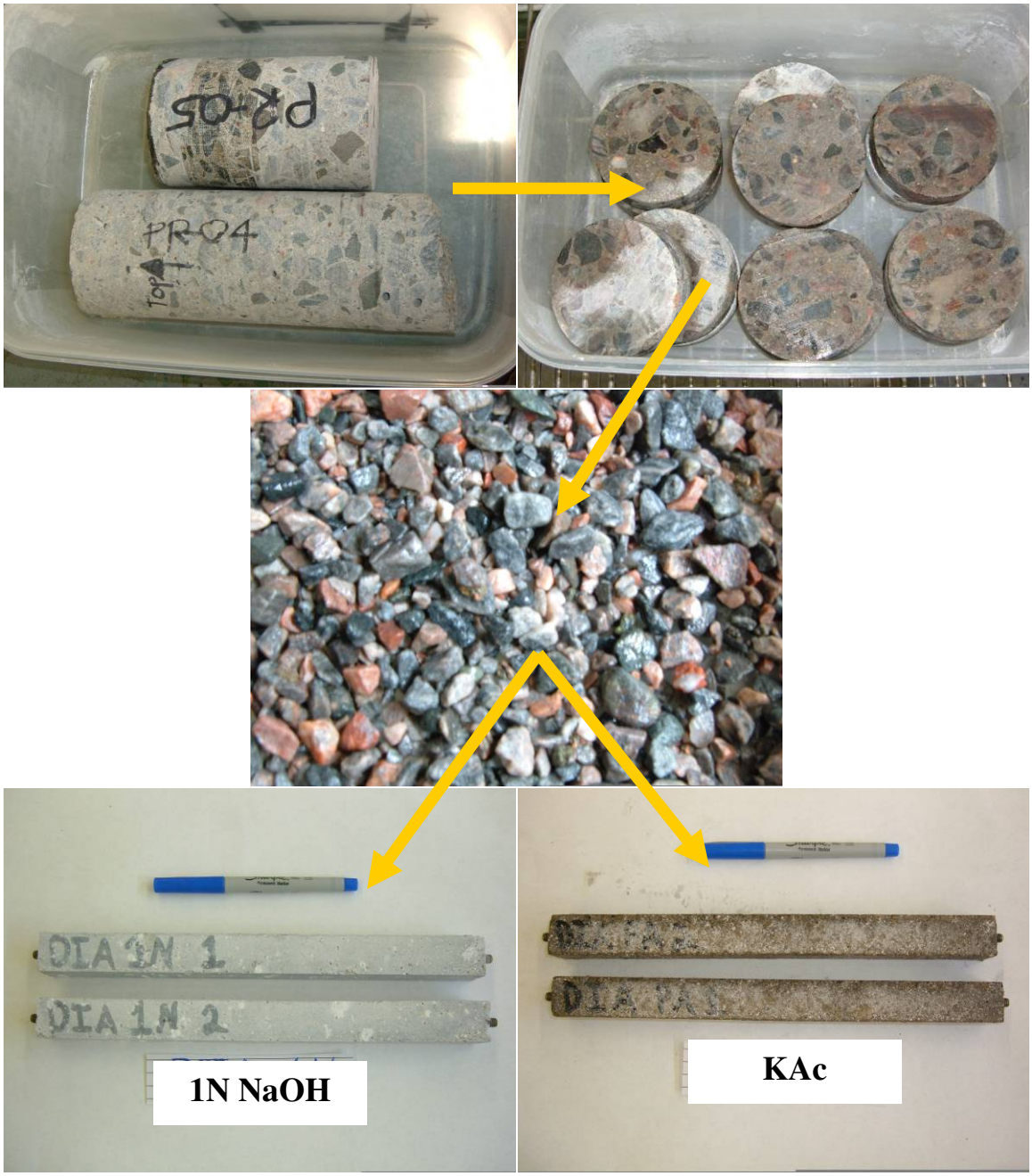

Figure B.5: Visual Progression of Aggregate Removal for Airport #2 Core Samples

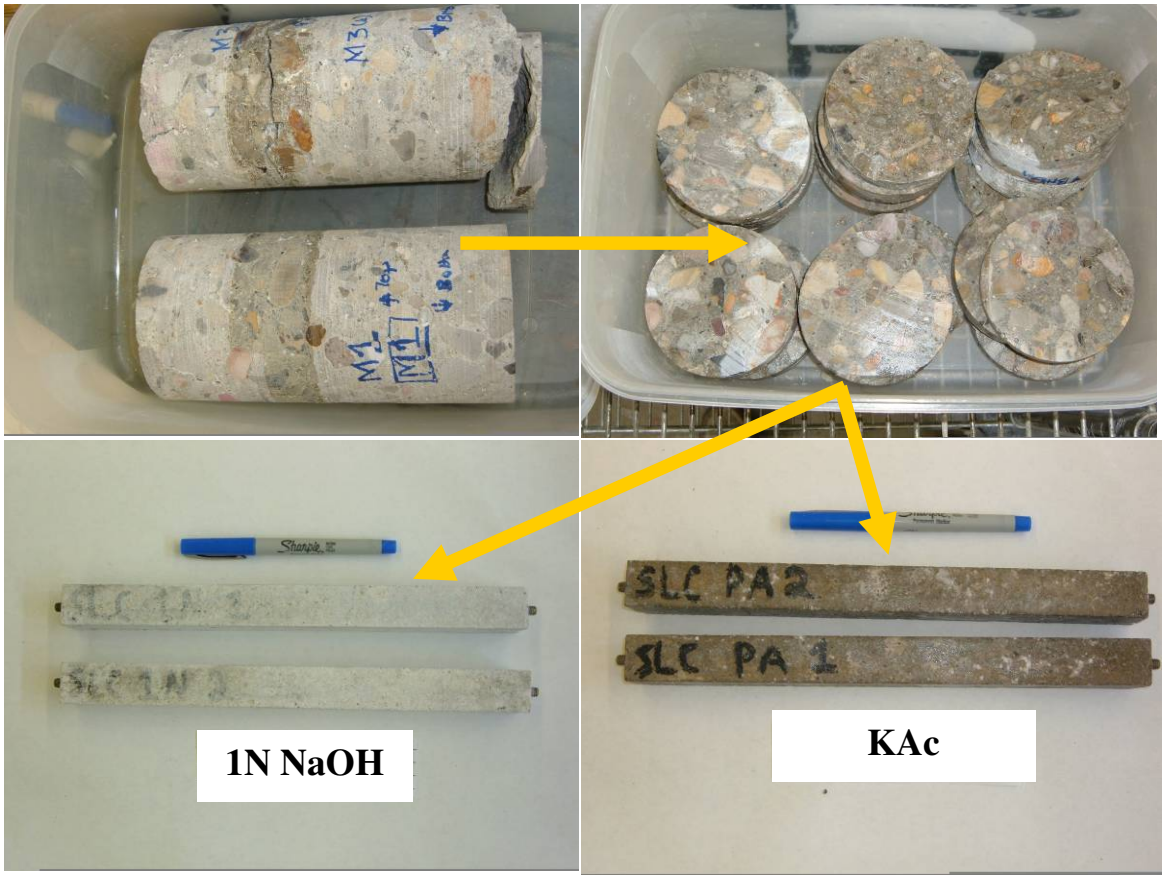

Figure B.6: Visual Progression of Aggregate Removal for Airport #1 Core Samples

C. SEM AND EDX OUTPUT FOR MORTAR BAR SAMPLES MADE WITH THE RETRIEVED AGGREGATE

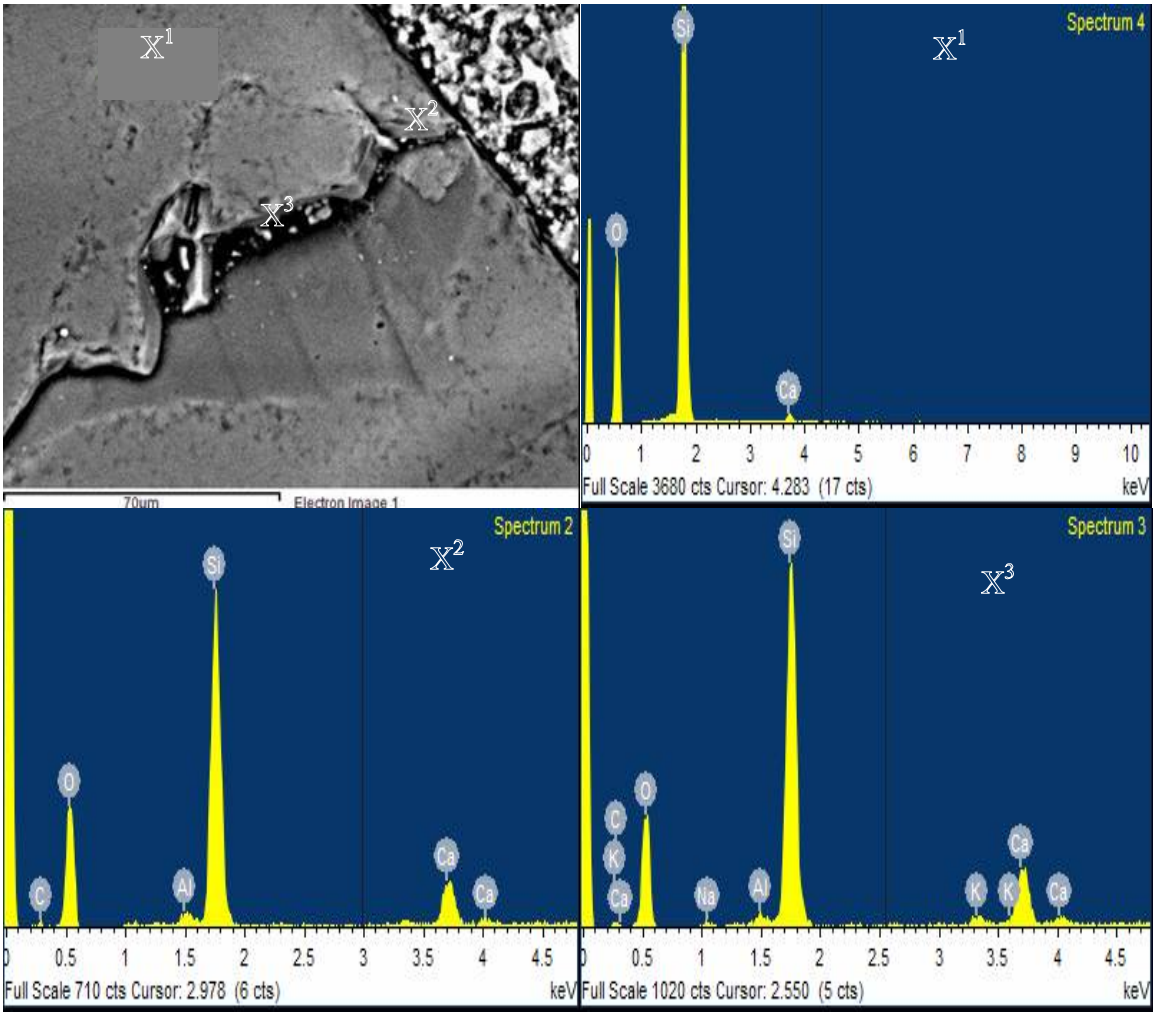

Figure C.1: SEM and EDX Output for NM-PA-1N Sample

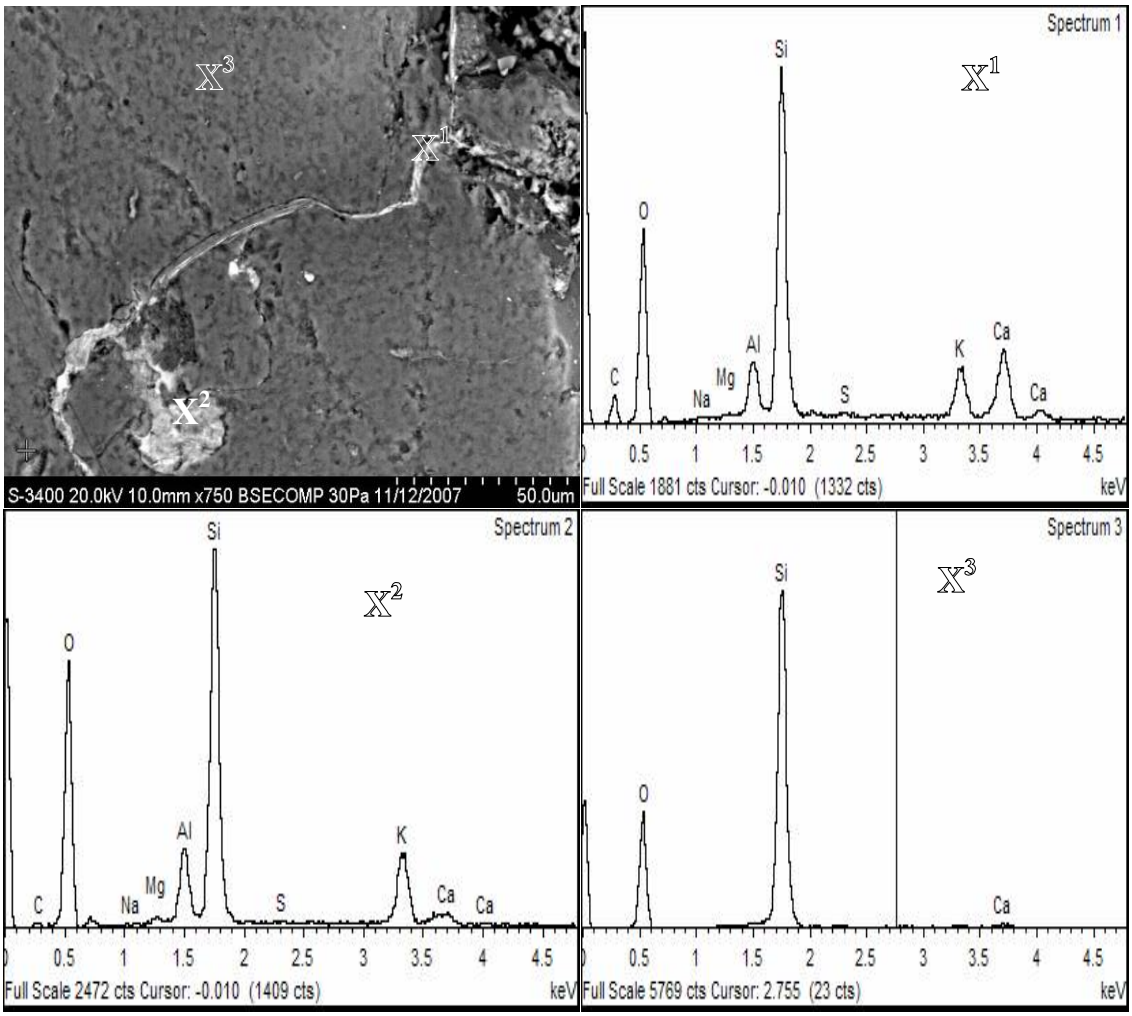

Figure C.2: SEM and EDX Output for NM-PA-PA Sample

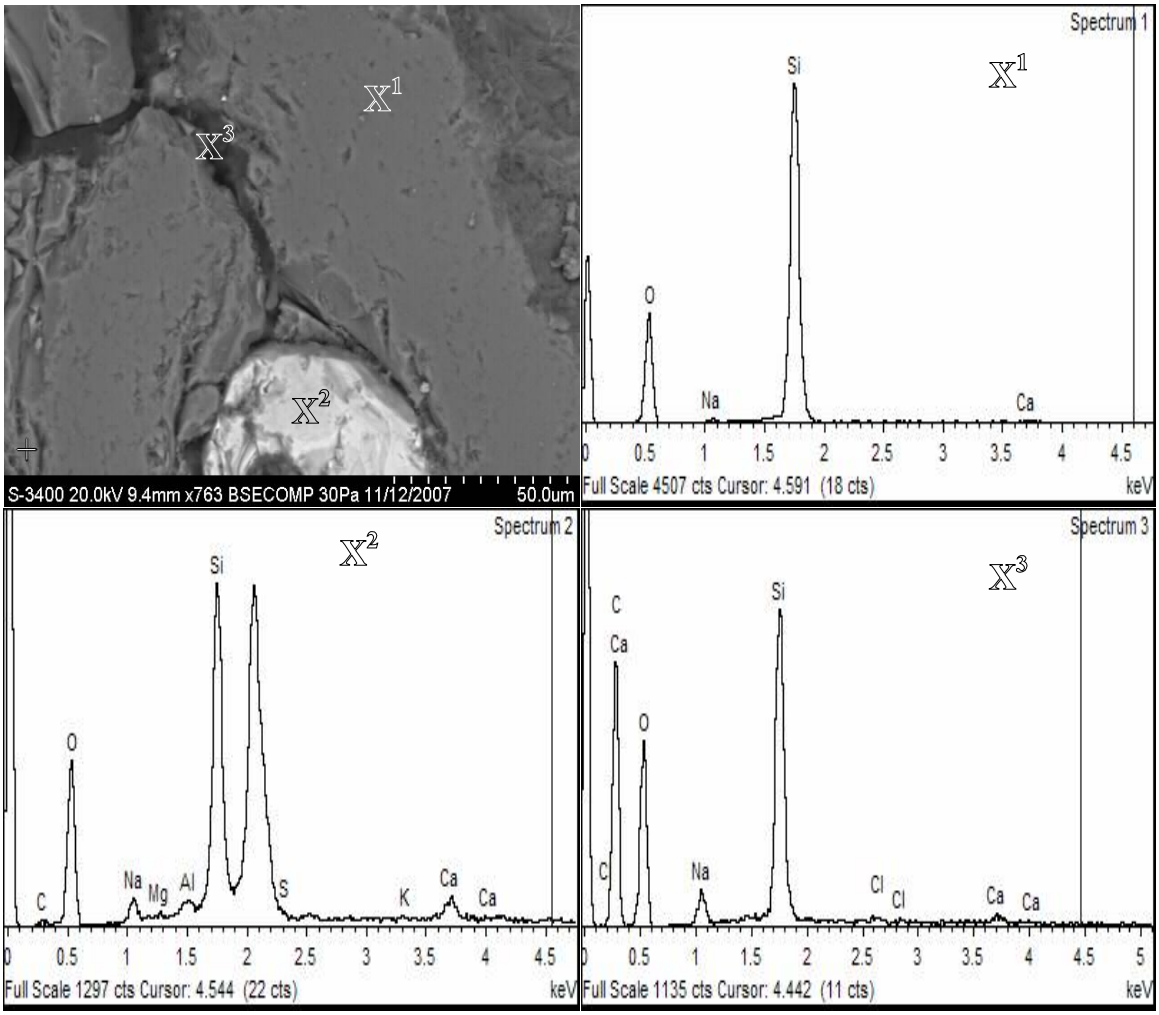

Figure C.3: SEM and EDX Output for SD-PA-1N Sample

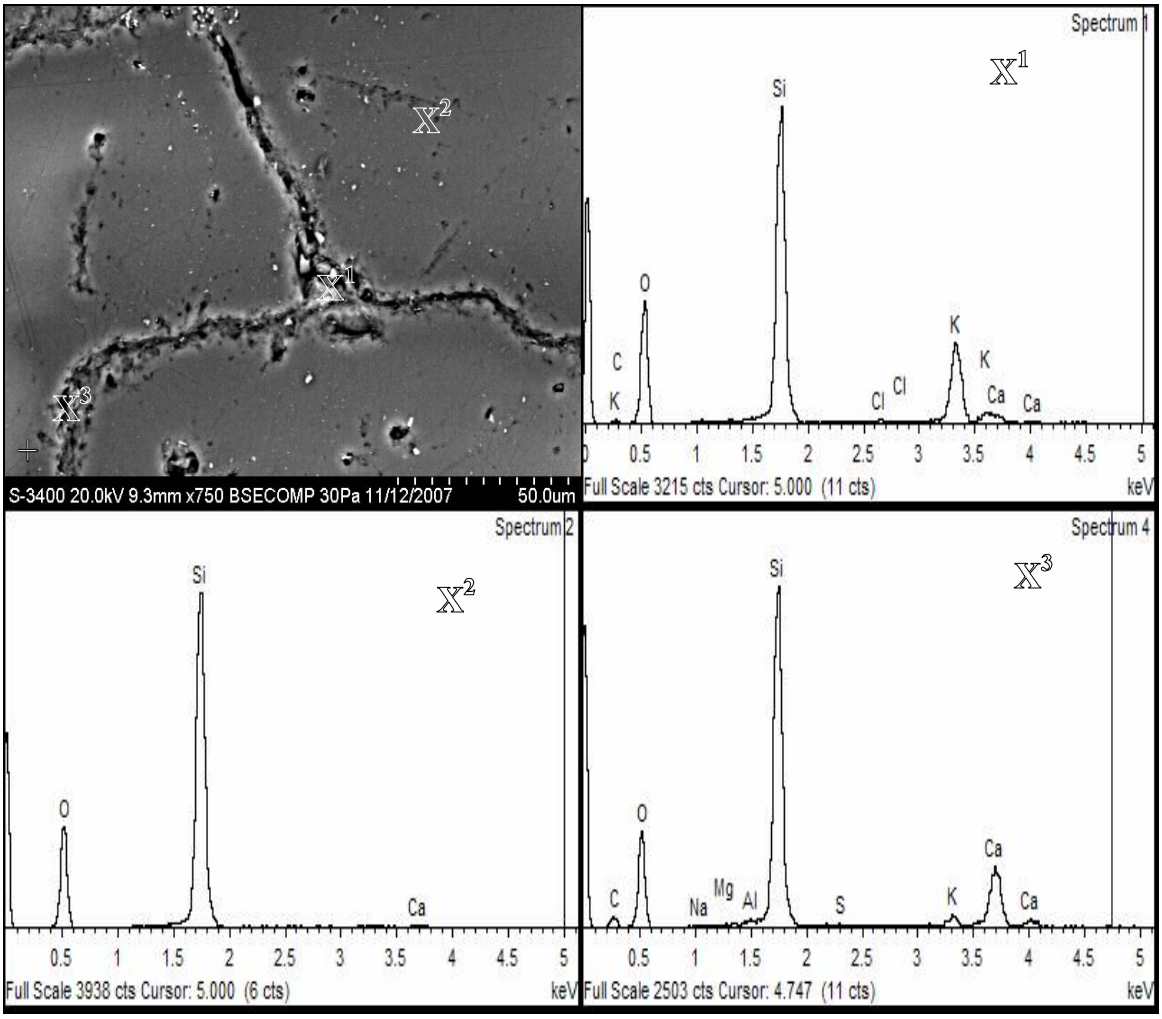

Figure C.4: SEM and EDX Output for SD-PA-PA Sample

D. ASTM C 1293(PRE-REMOVAL) AND ASTM C 1260 (PRE AND POST REMOVAL) AGGREGATE CLASSIFICATIONS

Table D.1: ASTM C 1293(Pre-Removal) and ASTM C 1260 (Pre and Post Removal) Aggregate Classifications

Aggregate	Test	Solution	Classification	Post Removal / Pre-Removal
NM	ASTM C 1293	Control	PD	Pre-Removal
NM	ASTM C 1293	1N NaOH	PD	Pre-Removal
NM	ASTM C 1293	PA	PD	Pre-Removal
NM	ASTM C 1260	1N NaOH	PD	Pre-Removal
NM	ASTM C 1260	PA	PD	Pre-Removal
NM	ASTM C 1260	PA-PA	PD	Post Removal
NM	ASTM C 1260	PA-1N	PD	Post Removal
NM	ASTM C 1260	1N-PA	PD	Post Removal
NM	ASTM C 1260	1N-1N	PD	Post Removal
SD	ASTM C 1293	Control	PD	Pre-Removal
SD	ASTM C 1293	1N NaOH	PD	Pre-Removal
SD	ASTM C 1293	PA	PD	Pre-Removal
SD	ASTM C 1260	1N NaOH	PD	Pre-Removal
SD	ASTM C 1260	PA	PD	Pre-Removal
SD	ASTM C 1260	PA-PA	PD	Post Removal
SD	ASTM C 1260	PA-1N	PI & PD	Post Removal
SD	ASTM C 1260	1N-PA	PI & PD	Post Removal
SD	ASTM C 1260	1N-1N	PI & PD	Post Removal
NC	ASTM C 1293	Control	PD	Pre-Removal
NC	ASTM C 1293	1N NaOH	PD	Pre-Removal
NC	ASTM C 1293	PA	PD	Pre-Removal
NC	ASTM C 1260	1N NaOH	PD	Pre-Removal
NC	ASTM C 1260	PA	PD	Pre-Removal
NC	ASTM C 1260	PA-PA	PD	Post Removal
NC	ASTM C 1260	PA-1N	PD	Post Removal
NC	ASTM C 1260	1N-PA	PD	Post Removal
NC	ASTM C 1260	1N-1N	PD	Post Removal
Airport #1	ASTM C 1260	1N NaOH	PD	Post Removal
Airport #1	ASTM C 1260	PA	PD	Post Removal
Airport #2	ASTM C 1260	1N NaOH	PI	Post Removal
Airport #2	ASTM C 1260	PA	PI & PD	Post Removal
PD = Potentially Deleterious Expansions (%Expansions > 0.2%)				
PI & PD = Potentially Innocous and Deleterious in Field Performance (0.1% < %Expansions < 0.2%)				
I = Innocuous Behavior in Most Cases (%Expansions < 0.1%)				

E. DATA FROM ASTM C 1260 (PRE-REMOVAL)

Table E.1: ASTM C 1260(Pre-Removal) Results for the New Mexico Aggregate

NEW MEXICO ASTM 1260 (1N NaOH)					
	comparator readings				
days	ref bar	bar1	bar2	bar3	bar4
0	0.01	0.2969	0.2752	0.332	0.3206
3	0.0036	0.3477	0.3264	0.3798	0.382
7	0.0036	0.3902	0.3705	0.4213	0.4095
10	0.0038	0.4128	0.3947	0.4439	0.4311
14	0.0036	0.434	0.417	0.4677	0.4521
21	0.0033	0.455	0.4387	0.4909	0.4725
28	0.0031	0.4693	0.4532	0.5064	0.4865
NEW MEXICO ASTM 1260 (POTASSIUM ACETATE)					
days	ref bar	bar1	bar2	bar3	bar4
0	0.01	0.3017	0.3135	0.325	0.3132
3	0.0036	0.4212	0.4293	0.4441	0.4299
7	0.0036	0.4519	0.4594	0.4747	0.4614
10	0.0038	0.4533	0.4605	0.476	0.4629
14	0.0036	0.4539	0.4612	0.4765	0.4635
21	0.0033	0.4541	0.4618	0.477	0.4639
28	0.0031	0.4554	0.4628	0.4781	0.4652

Table E.2: ASTM C 1260(Pre-Removal) Results for the South Dakota Aggregate

SOUTH DAKOTA ASTM 1260 (1N NaOH)					
	comparator readings				
days	ref bar	bar1	bar2	bar3	bar4
0	0.1402	0.454	0.4434	0.424	0.4514
3	0.14	0.4578	0.4472	0.4279	0.4553
7	0.1404	0.4659	0.4553	0.4356	0.463
10	0.1406	0.4717	0.4611	0.4409	0.4684
14	0.141	0.4773	0.4677	0.4472	0.4748
21	0.0754	0.4222	0.4128	0.3914	0.4193
28	0.0118	0.3668	0.3577	0.3361	0.3639
SOUTH DAKOTA ASTM 1260 (POTASSIUM ACETATE)					
days	ref bar	bar1	bar2	bar3	bar4
0	0.1402	0.4261	0.415	0.44	0.4288
3	0.14	0.4298	0.4186	0.4438	0.4326
7	0.1404	0.4444	0.4327	0.4575	0.4469
10	0.1406	0.4563	0.445	0.4708	0.4593
14	0.141	0.4645	0.4536	0.4802	0.4687
21	0.0754	0.4025	0.3919	0.4189	0.4069
28	0.0118	0.3408	0.3301	0.3573	0.3452

Table E.3: ASTM C 1260(Pre-Removal) Results for the North Carolina Aggregate

NORTH CAROLINA ASTM 1260 (1N NaOH)					
	comparator readings				
days	ref bar	bar1	bar2	bar3	bar4
0	0.14	0.3767	0.4485	0.4548	0.4315
3	0.1401	0.3892	0.4614	0.4675	0.4446
7	0.1403	0.4105	0.4831	0.4899	0.4675
10	0.1403	0.4189	0.4915	0.4982	0.4757
14	0.1402	0.4273	0.5002	0.5072	0.4847
21	0.1406	0.4402	0.5108	TALL	0.4975
28	0.011	0.3215	0.3942	0.4031	0.3801
NORTH CAROLINA ASTM 1260 (POTASSIUM ACETATE)					
days	ref bar	bar1	bar2	bar3	bar4
0	0.14	0.447	0.4478	0.412	0.4209
3	0.1401	0.4516	0.4523	0.4162	0.4254
7	0.1403	0.4847	0.4879	0.45	0.4591
10	0.1403	0.5032	0.5027	0.4634	0.4727
14	0.1402	0.5062	0.5061	0.4666	0.4761
21	0.1406	0.5091	0.5093	0.4692	0.4788
28	0.011	0.3813	0.3817	0.3416	0.3511

F. DATA FROM ASTM C 1293 (PRE-REMOVAL)

Table F.1: ASTM C 1293(Pre-Removal) Results for the New Mexico Aggregate

NEW MEXICO ASTM 1293 (CONTROL)				
days	**ref bar**	**bar1**	**bar2**	**bar3**
0	0.0019	0.3577	0.3538	0.3637
7	0.0006	0.356	0.3531	0.3627
28	0.0038	0.3636	0.3605	0.3694
56	0.004	0.3637	0.3613	0.3708
90	0.0015	0.3688	0.3666	0.375
120	0.0151	0.3857	0.3844	0.3915
150	0.0045	0.3757	0.375	0.3821
180	0.0056	0.3787	0.3783	0.3853
270	0.0055	0.3785	0.3784	0.385
360	0.0058	0.3799	0.3799	0.3859
NEW MEXICO ASTM 1293 (1N NaOH)				
days	**ref bar**	**bar1**	**bar2**	**bar3**
0	0.0019	0.3723	0.3444	0.3478
7	0.0006	0.3724	0.3441	0.3475
28	0.0038	0.3803	0.3518	0.3547
90	0.0015	0.3869	0.36	0.3605
120	0.0151	0.4037	0.3776	0.3767
150	0.0045	0.3943	0.368	0.3672
180	0.0056	0.3978	0.3731	0.3706
270	0.0055	0.402	0.3773	0.3742
360	0.0058	0.4073	0.3838	0.3784
NEW MEXICO ASTM 1293 (POTASSIUM ACETATE)				
days	**ref bar**	**bar1**	**bar2**	**bar3**
0	0.0019	0.344	0.3194	0.3677
7	0.0006	0.3444	0.3197	0.3672
28	0.0038	0.361	0.3363	0.3842
90	0.0015	0.4144	0.3966	0.4399
120	0.0151	0.5147	0.4951	0.5156
150	badly cracked, cant take readings			

Table F.2: ASTM C 1293(Pre-Removal) Results for the South Dakota Aggregate

SOUTH DAKOTA ASTM 1293 (CONTROL)				
days	**ref bar**	**bar1**	**bar2**	**bar3**
0	0.0008	0.3325	0.3235	0.3521
7	0.0016	0.331	0.3232	0.352
28	0.0027	0.3332	0.3259	0.3542
56	0.0024	0.3348	0.3273	0.3559
90	0.0021	0.3351	0.3276	0.3562
120	0.0058	0.3406	0.3321	0.3602
150	0.0064	0.343	0.334	0.3629
180	0.0055	0.3436	0.3344	0.3635
270	0.0048	0.345	0.336	0.3646
360	0.006	0.3488	0.3395	0.3682
SOUTH DAKOTA ASTM 1293 (1N NaOH)				
days	**ref bar**	**bar1**	**bar2**	**bar3**
0	0.0008	0.3469	0.3428	0.3374
7	0.0019	0.3481	0.3436	0.3381
28	0.0027	0.3499	0.3453	0.3405
56	0.0024	0.3503	0.346	0.3411
90	0.0021	0.3516	0.347	0.3423
120	0.0058	0.3563	0.3521	0.3474
150	0.0064	0.358	0.3536	0.3489
180	0.0055	0.3582	0.3539	0.3493
270	0.0048	0.359	0.3548	0.3501
360	0.006	0.3621	0.3577	0.3531
SOUTH DAKOTA ASTM 1293 (POTASSIUM ACETATE)				
days	**ref bar**	**bar1**	**bar2**	**bar3**
0	0.0008	0.2956	0.3498	0.4532
7	0.0019	0.2963	0.35	0.4541
28	0.0027	0.2978	0.3516	0.4556
56	0.0024	0.2979	0.3517	0.4557
90	0.0021	0.2981	0.3518	0.456
120	0.0058	0.3022	0.356	0.4601
150	0.0064	0.3033	0.3572	0.4614
180	0.0055	0.3037	0.3577	0.4616
270	0.0048	0.3064	0.3602	0.465
360	0.006	0.3157	0.3696	0.475

Table F.3: ASTM C 1293(Pre-Removal) Results for the North Carolina Aggregate

NORTH CAROLINA ASTM 1293 (CONTROL)				
days	**ref bar**	**bar1**	**bar2**	**bar3**
0	0.0017	0.3393	0.278	0.2701
7	0.0016	0.3387	0.2773	0.2692
28	0.003	0.3418	0.2801	0.2724
56	0.0023	0.3468	0.2801	0.2724
90	0.0021	0.3531	0.2896	0.2842
120	0.0055	0.3601	0.2961	0.2905
180	0.0065	0.3642	0.3006	0.2942
270	0.0048	0.3654	0.3012	0.2947
360	0.0055	0.3685	0.3039	0.2979
NORTH CAROLINA ASTM 1293 (1N NaOH)				
	ref bar	**bar1**	**bar2**	**bar3**
0	0.0017	0.3048	0.3422	0.3108
7	0.0019	0.3049	0.3427	0.3108
28	0.003	0.3066	0.3445	0.3126
56	0.0023	0.3092	0.3482	0.315
90	0.0021	0.3134	0.3528	0.3191
120	0.0055	0.3202	0.3599	0.3263
150	0.0065	0.3231	0.3628	0.3296
180	0.0063	0.3239	0.3636	0.3308
270	0.0048	0.3248	0.3643	0.3324
360	0.0055	0.3282	0.3673	0.336
NORTH CAROLINA ASTM 1293 (POTASSIUM ACETATE)				
	ref bar	**bar1**	**bar2**	**bar3**
0	0.0017	0.3572	0.3287	0.3543
7	0.0019	0.3575	0.3294	0.3541
28	0.003	0.3588	0.3304	0.3554
56	0.0023	0.3604	0.3324	0.3571
90	0.0021	0.3671	0.3388	0.3627
120	0.0055	0.3784	0.3494	0.3735
150	0.0065	0.3881	0.359	0.3824
180	0.0063	0.397	0.3685	0.3909
270	0.0048	0.4247	0.3961	0.4178
360	0.0055	0.4649	0.44	0.4555

G. DATA FROM ASTM C 1260 (POST REMOVAL)

Table G.1: ASTM C 1260(Post Removal) Results for the New Mexico Aggregate

Referance (in.)	Date	NM-PA-PA-1	NM-PA-PA-2	NM-PA-PA-3	NM-PA-1N-1	NM-PA-1N-2	NM-PA-1N-3
0.0097	17-Jan	0.3065	0.3093	0.2914	0.2940	0.2962	0.3268
0.0103	18-Jan	0.3159	0.3185	0.3006	0.3030	0.3049	0.3353
0.0100	21-Jan	0.4078	0.4093	0.3937	0.3432	0.3421	0.3755
0.0100	25-Jan	0.4269	0.4271	0.4120	0.3589	0.3569	0.3921
0.0095	28-Jan	0.4282	0.4283	0.4134	0.3620	0.3602	0.3956
0.0100	1-Feb	0.4320	0.4319	0.4168	0.3704	0.3678	0.4055
0.0102	8-Feb	0.4337	0.4335	0.4186	0.3773	0.3744	0.4124
0.0099	15-Feb	0.4341	0.4343	0.4193	0.3826	0.3796	0.4182
Referance (in.)	**Date**	**NM-1N-PA-1**	**NM-1N-PA-2**	**NM-1N-PA-3**	**NM-1N-1N-1**	**NM-1N-1N-2**	**NM-1N-1N-3**
0.0068	9-Aug	0.2976	0.3037	0.3066	0.2845	0.2909	0.3242
0.0069	10-Aug	0.3047	0.3105	0.3115	0.2911	0.2976	0.3303
0.0067	13-Aug	0.3728	0.3794	0.3851	0.3262	0.3319	0.3648
0.0072	17-Aug	0.4030	0.4087	0.4140	0.3556	0.3600	0.3927
0.0071	20-Aug	0.4065	0.4121	0.4178	0.3653	0.3691	0.4024
0.0072	24-Aug	0.4092	0.4149	0.4220	0.3758	0.3791	0.4121
0.0075	1-Sep	0.4116	0.4173	0.4246	0.3856	0.3883	0.4217
0.0071	8-Sep	0.4124	0.4182	0.4241	0.3927	0.3952	0.4292

Table G.2: ASTM C 1260(Post Removal) Results for the South Dakota Aggregate

Referance (in.)	Date	SD-PA-PA-1	SD-PA-PA-2	SD-PA-PA-3	SD-PA-1N-1	SD-PA-1N-2	SD-PA-1N-3
0.0097	17-Jan	0.3031	0.2859	0.3663	0.3068	0.2999	0.3051
0.0103	18-Jan	0.3116	0.2945	0.3745	0.3152	0.3084	0.3133
0.0100	21-Jan	0.3162	0.2992	0.3794	0.3175	0.3108	0.3153
0.0100	25-Jan	0.3233	0.3063	0.3867	0.3231	0.3166	0.3211
0.0095	28-Jan	0.3251	0.3082	0.3889	0.3235	0.3163	0.3208
0.0100	1-Feb	0.3302	0.3130	0.3942	0.3270	0.3202	0.3242
0.0102	8-Feb	0.3317	0.3142	0.3954	0.3300	0.3231	0.3274
0.0099	15-Feb	0.3323	0.3151	0.3963	0.3315	0.3251	0.3295
Referance (in.)	**Date**	**SD-1N-PA-1**	**SD-1N-PA-2**	**SD-1N-PA-3**	**SD-1N-1N-1**	**SD-1N-1N-2**	**SD-1N-1N-3**
0.0068	9-Aug	0.2999	0.2852	0.2997	0.2929	0.2939	0.2789
0.0069	10-Aug	0.3078	0.2926	0.3068	0.3008	0.3020	0.2860
0.0067	13-Aug	0.3105	0.2958	0.3098	0.3024	0.3036	0.2878
0.0072	17-Aug	0.3162	0.3015	0.3162	0.3089	0.3103	0.2952
0.0071	20-Aug	0.3199	0.3054	0.3194	0.3128	0.3143	0.2991
0.0072	24-Aug	0.3247	0.3098	0.3237	0.3175	0.3200	0.3044
0.0075	1-Sep	0.3273	0.3124	0.3262	0.3254	0.3277	0.3126
0.0071	8-Sep	0.3281	0.3128	0.3267	0.3313	0.3330	0.3176

Table G.3: ASTM C 1260(Post Removal) Results for the North Carolina Aggregate

Referance (in.)	Date	NC-PA-PA-1	NC-PA-PA-2	NC-PA-PA-3	NC-PA-1N-1	NC-PA-1N-2	NC-PA-1N-3
0.0097	27-Feb	0.2947	0.2827	0.2828	0.3127	0.2879	0.2737
0.0081	28-Feb	0.3022	0.2900	0.2900	0.3199	0.2952	0.2810
0.0087	3-Mar	0.3141	0.3023	0.3018	0.3403	0.3157	0.2999
0.0098	7-Mar	0.3524	0.3428	0.3375	0.3568	0.3316	0.3149
0.0094	10-Mar	0.3582	0.3486	0.3430	0.3650	0.3395	0.3223
0.0087	14-Mar	0.3594	0.3502	0.3441	0.3725	0.3465	0.3287
0.0092	21-Mar	0.3624	0.3532	0.3472	0.3840	0.3584	0.3399
0.0090	28-Mar	0.3636	0.3546	0.3483	0.3923	0.3663	0.3472
Referance (in.)	**Date**	**NC-1N-PA-1**	**NC-1N-PA-2**	**NC-1N-PA-3**	**NC-1N-1N-1**	**NC-1N-1N-2**	**NC-1N-1N-3**
0.0068	9-Aug	0.2446	0.3093	0.3110	0.3243	0.2530	0.3039
0.0069	10-Aug	0.2513	0.3161	0.3176	0.3313	0.2596	0.3107
0.0067	13-Aug	0.2558	0.3202	0.3223	0.3404	0.2688	0.3197
0.0072	17-Aug	0.2792	0.3420	0.3457	0.3610	0.2895	0.3403
0.0071	20-Aug	0.2929	0.3545	0.3598	0.3706	0.2992	0.3500
0.0072	24-Aug	0.2977	0.3590	0.3646	0.3820	0.3104	0.3614
0.0075	1-Sep	0.2998	0.3611	0.3669	0.3938	0.3224	0.3734
0.0071	8-Sep	0.2997	0.3616	0.3670	0.4028	0.3315	0.3824

Table G.4: ASTM C 1260(Post Removal) Results for Airport #1 Cores

Referance (in.)	Date	SLC PA 1	SLC PA 2	SLC 1N 1	SLC 1N 2
0.0080	19-Jul	0.3234	0.2888	0.3320	0.3048
0.0073	20-Jul	0.3306	0.2960	0.3394	0.3126
0.0078	25-Jul	0.3379	0.3033	0.3495	0.3240
0.0101	27-Jul	0.3456	0.3116	0.3592	0.3329
0.0100	31-Jul	0.3511	0.3175	0.3680	0.3420
0.0099	3-Aug	0.3529	0.3198	0.3737	0.3478
0.0099	7-Aug	0.3540	0.3212	0.3814	0.3548
0.0098	10-Aug	0.3548	0.3216	0.3860	0.3598
0.0105	12-Aug	0.3555	0.3225	0.3894	0.3637
0.0098	18-Aug	0.3551	0.3220	0.3972	0.3708

Table G.5: ASTM C 1260(Post Removal) Results for Airport #2 Cores

Referance (in.)	Date	DIA PA 1	DIA PA 2	DIA 1N 1	DIA 1N 2
0.0080	19-Jul	0.3164	0.3245	0.3201	0.3205
0.0073	20-Jul	0.3225	0.3306	0.3263	0.3266
0.0078	25-Jul	0.3272	0.3352	0.3302	0.3305
0.0101	27-Jul	0.3321	0.3401	0.3344	0.3349
0.0100	31-Jul	0.3366	0.3440	0.3355	0.3366
0.0099	3-Aug	0.3386	0.3462	0.3370	0.3370
0.0099	7-Aug	0.3408	0.3481	0.3381	0.3381
0.0098	10-Aug	0.3419	0.3499	0.3385	0.3392
0.0105	12-Aug	0.3432	0.3510	0.3398	0.3402
0.0098	18-Aug	0.3436	0.3517	0.3395	0.3404

H. CONCRETE MIX DESIGNS

MATERIALS

Material	Type	Supplier	SSDSG	Abs%	Free%	Other Data
Type I Cement	Type I	Lafarge	3.15			
RHA		El Paso, TX	2.08			
Clemson Sand	Natural	Beside Lowry	2.58	2.00%	0.00%	FM=2.91
Clemson Stone	#67	Beside Lowry	2.56	1.10%	0.00%	DRUW=96.6pcf
GL3030	HRWR	MasterBuilders	1.20	70%	Water	10.00 lbs/gal
Clemson Water	Tap	CWS	1.00			

MIX PROPORTIONS

Material	Design Quantities: Dry Weights	Design Quantities: SSD Weights	Design Quantities: SSD Volume	Mixer Setup Quantities	/cy	/batch
Type I Cement	590 lbs/cy	***590*** lbs/cy	3.00 cf/cy	590.0 lbs/cy	590.00 lbs	9.83 lbs
RHA	65 lbs/cy	***65*** lbs/cy	0.50 cf/cy	65.0 lbs/cy	65.00 lbs	1.08 lbs
Clemson Sand	1112 lbs/cy	***1135*** lbs/cy	7.05 cf/cy	1135.0 lbs/cy	1135.00 lbs	18.92 lbs
Clemson Stone	1623 lbs/cy	***1641*** lbs/cy	10.27 cf/cy	1641.0 lbs/cy	1641.00 lbs	27.35 lbs
GL3030	1.07 lbs/cy	3.58 lbs/cy	0.05 cf/cy	45.85 oz/cy	45.85 oz	21.66 gms
Clemson Water	340.00 lbs/cy	***340.00*** lbs/cy	5.45 cf/cy	340.0 lbs/cy	380.75 lbs	6.35 lbs
Air @ **2.5%**			0.68 cf/cy			
SSD Totals		3774.58 lbs/cy	27.00 cf/cy			

Design Notes:

FA Volume / Aggregate Volume =	0.41
CA Volume / Aggregate Volume =	0.59
FA/C =	1.92
CA/C =	2.78
Paste Volume / Total Volume =	0.36
Mortar Volume / Total Volume =	0.60
W/CM with Admix =	0.523
W/C =	0.576
W/CM =	0.519
Plastic Density [pcf] =	139.8
Oven Dry Density, Oc [pcf] =	130.4 (Aggregate Dry Weight + Cement Weight x 1.2) / (Volume)
Equilibrium Density, Ec [pcf] =	133.4 Oc + 3pcf
Design Strength, f'c [psi] =	4500

Admixture Dosage Rates		
GL3030	***7.0***	oz/cwt

Batch Size =	**0.45**	cf
	0.017	cy

10% RHA Mix Design

Figure H.1: 10% RHA Concrete Mix Design

MATERIALS

Material	Type	Supplier	Current $		SSDSG	Abs%	Free%	Other Data
Type I Cement	Type I	Lafarge	$105.00	/Ton	3.15			
RHA		El Paso, TX	$40.00	/Ton	2.08			
Clemson Sand	Natural	Beside Lowry	$12.00	/Ton	2.58	2.00%	0.00%	FM=2.91
Clemson Stone	#67	Beside Lowry	$15.00	/Ton	2.56	1.10%	0.00%	DRUW=96.6pcf
GL3030	HRWR	MasterBuilders	$7.00	/Gallor	1.20	70%	Water	10.00 lbs/gal
Clemson Water	Tap	CWS	$0.00		1.00			

MIX PROPORTIONS

Material	Design Quantities: Dry Weights	SSD Weights	SSD Volume	Mixer Setup Quantities	/cy	/batch
Type I Cement	655 lbs/cy	***655*** lbs/cy	3.33 cf/cy	655.0 lbs/cy	655.00 lbs	10.92 lbs
RHA						
Clemson Sand	1123 lbs/cy	***1146*** lbs/cy	7.12 cf/cy	1146.0 lbs/cy	1146.00 lbs	19.10 lbs
Clemson Stone	1642 lbs/cy	***1660*** lbs/cy	10.39 cf/cy	1660.0 lbs/cy	1660.00 lbs	27.67 lbs
GL3030	0.38 lbs/cy	1.28 lbs/cy	0.02 cf/cy	16.38 oz/cy	16.38 oz	7.74 gms
Clemson Water	340.00 lbs/cy	***340.00*** lbs/cy	5.45 cf/cy	340.0 lbs/cy	381.18 lbs	6.35 lbs
Air @ **2.5%**			0.68 cf/cy			
SSD Totals		3802.28 lbs/cy	26.98 cf/cy			

Design Notes:

FA Volume / Aggregate Volume =	0.41
CA Volume / Aggregate Volume =	0.59
FA/C =	1.75
CA/C =	2.53
Paste Volume / Total Volume =	0.35
Mortar Volume / Total Volume =	0.61
W/CM with Admix =	0.520
W/C =	0.519
W/CM =	0.519
Plastic Density [pcf] =	140.9
Oven Dry Density, Oc [pcf] =	131.6
Equilibrium Density, Ec [pcf] =	134.6
Design Strength, f'c [psi] =	4500

(Aggregate Dry Weight + Cement Weight x 1.2) / (Volume)

Oc + 3pcf

Admixture Dosage Rates		
GL3030	***2.5***	oz/cwt

Batch Size =	**0.45**	cf
	0.017	cy

Control Mix Design to Compare to:
10% RHA Mix Design

Figure H.2: Control RHA Concrete Mix Design

I. DATA FROM THE STANDARD AND MODIFIED ASTM C 1202

Table I.1: Modified ASTM C 1202 Results for the Control Samples

	Coulombs					
	14 Day	**14 Day**	**14 Day Avg**	**28-Day**	**28-Day**	**28-Day Avg**
Time (min)	**Control**	**Control**	**Control**	**Control**	**Control**	**Control**
1	19	4	12	15	14	15
30	673	635	654	556	513	535
60	1614	1315	1465	1254	1143	1199
90	3128	2286	2707	2074	1870	1972
110	4047	2916	3482			
120	4549	3394	3972	3006	2678	2842
135	5000	3997	4499	3513	3109	3311
150				4050	3557	3804
155						
165				4627	4024	4326
170						
180						
185						
210						
225						
240						
270						
300						
330						
360						

Table I.2: Modified ASTM C 1202 Results for the 5% RHA Samples

	Coulombs					
	14 Day	**14 Day**	**14 Day Avg**	**28-Day**	**28-Day**	**28-Day Avg**
Time (min)	**5%**	**5%**	**5%**	**5%**	**5%**	**5%**
1	14	16	15	11	11	11
30	482	554	518	385	373	379
60	1069	1229	1149	840	800	820
90	1755	2009	1882	1362	1281	1322
110						
120	2529	2879	2704	1946	1813	1880
135				2261	2097	2179
150	3394	3832	3613	2590	2391	2491
155	3548	3999	3774			
165				2934	2697	2816
170	4084	4300	4192			
180				3292	3013	3153
185						
210				4052	3674	3863
225				4463	4020	4242
240						
270						
300						
330						
360						

Table I.3: Modified ASTM C 1202 Results for the 10% RHA Samples

	Coulombs			
	14 Day	**14 Day**	**14 Day Avg**	**28-Day**
Time (min)	**10%**	**10%**	**10%**	**10%**
1	5	6	6	4
30	212	202	207	152
60	552	428	490	318
90	1062	673	868	497
110				
120	1655	935	1295	686
135				785
150	2535	1214	1875	886
155				
165				989
170				
180	3915	1507	2711	1095
185	4175			
210		1811		1312
225				
240		2126		1536
270		2450		1767
300		2728		2004
330		3121		2248
360		3465		2496

Table I.4: Modified ASTM C 1202 Results for the 15% RHA Samples

	Coulombs					
	14 Day	**14 Day**	**14 Day Avg**	**28-Day**	**28-Day**	**28-Day Avg**
Time (min)	**15%**	**15%**	**15%**	**15%**	**15%**	**15%**
1	4	3	4	3	3	3
30	145	123	134	92	92	92
60	301	254	278	189	190	190
90	466	395	431	291	292	292
110						
120	637	542	590	396	398	397
135				450	453	452
150	812	695	754	505	508	507
155						
165				561	564	563
170						
180	992	853	923	617	620	619
185						
210	1173	1015	1094	731	735	733
225				789	793	791
240	1355	1180	1268	848	852	850
270	1538	1348	1443	966	971	969
300	1721	1519	1620	1087	1093	1090
330	1904	1692	1798	1208	1216	1212
360	2085	1866	1976	1332	1340	1336

Table I.5: Modified ASTM C 1202 Results for the 20% RHA Samples

	Coulombs					
	14 Day	**14 Day**	**14 Day Avg**	**28-Day**	**28-Day**	**28-Day Avg**
Time (min)	**20%**	**20%**	**20%**	**20%**	**20%**	**20%**
1	2	2	2	29	29	29
30	89	74	82	58	59	59
60	182	152	167	119	121	120
90	278	232	255	181	185	183
110						
120	376	314	345	245	250	248
135						
150	476	399	438	310	315	313
155						
165				343	349	346
170						
180	576	485	531	376	382	379
185						
210	677	573	625	443	449	446
225				476	483	480
240	779	662	721	510	517	514
270	881	752	817	578	585	582
300	982	842	912	647	653	650
330	1084	933	1009	716	722	719
360	1185	1023	1104	785	790	788

J. MODIFIED ASTM C 1202 DATA FROM STANDARD AND MODIFIED ASTM C 1567

Table J.1: Standard and Modified ASTM C 1567 Results for the Control Samples

Test 1								
Referance (in.)	**Date**	**0-PA-1**	**0-PA-2**	**0-PA-3**	**0-1N-1**	**0-1N-2**	**0-1N-3**	**Day**
0.0101	30-Jan	0.2838	0.2764	0.2796	0.2644	0.2804	0.2914	0
0.0100	31-Jan	0.2900	0.2826	0.2857	0.2711	0.2867	0.2978	0
0.0101	3-Feb	0.3007	0.2932	0.2961	0.2858	0.3014	0.3129	3
0.0102	7-Feb	0.3249	0.3161	0.3196	0.3030	0.3188	0.3313	7
0.0093	11-Feb	0.3348	0.3256	0.3293	0.3113	0.3268	0.3400	11
0.0098	14-Feb	0.3402	0.3315	0.3349	0.3173	0.3329	0.3465	14
0.0087	18-Feb	0.3503	0.3411	0.3447	0.3292	0.3447	0.3591	21
0.0096	25-Feb	0.3636	0.3528	0.3572	0.3448	0.3597	0.3754	28
Test 2								
Referance (in.)	**Date**	**0-PA-1**	**0-PA-2**	**0-PA-3**	**0-1N-1**	**0-1N-2**	**0-1N-3**	**Day**
0.0097	27-Feb	0.2584	0.2903	0.3003	0.3131	0.2860	0.3067	0
0.0081	28-Feb	0.2633	0.2953	0.3051	0.3182	0.2909	0.3116	0
0.0087	3-Mar	0.2812	0.3130	0.3230	0.3396	0.3130	0.3336	3
0.0098	7-Mar	0.3083	0.3404	0.3488	0.3573	0.3300	0.3498	7
0.0094	10-Mar	0.3169	0.3490	0.3574	0.3655	0.3380	0.3577	11
0.0087	14-Mar	0.3247	0.3570	0.3657	0.3735	0.3459	0.3658	14
0.0092	21-Mar	0.3408	0.3720	0.3805	0.3904	0.3628	0.3832	21
0.0090	28-Mar	0.3554	0.3867	0.3943	0.4067	0.3810	0.4007	28

Table J.2: Standard and Modified ASTM C 1567 Results for the 5% RHA Samples

Test 1								
Referance (in.)	**Date**	**5-PA-1**	**5-PA-2**	**5-PA-3**	**5-1N-1**	**5-1N-2**	**5-1N-3**	**Day**
0.0101	30-Jan	0.3210	0.2645	0.2936	0.2618	0.2765	0.2695	0
0.0100	31-Jan	0.3279	0.2714	0.3006	0.2688	0.2834	0.2764	0
0.0101	3-Feb	0.3400	0.2842	0.3118	0.2834	0.2982	0.2908	3
0.0102	7-Feb	0.3652	0.3089	0.3367	0.3004	0.3158	0.3081	7
0.0093	11-Feb	0.3756	0.3206	0.3478	0.3095	0.3248	0.3172	11
0.0098	14-Feb	0.3818	0.3245	0.3528	0.3158	0.3310	0.3234	14
0.0087	21-Feb	0.3882	0.3307	0.3591	0.3293	0.3440	0.3367	21
0.0096	28-Feb	0.3915	0.3339	0.3624	0.3463	0.3592	0.3528	28
Test 2								
Referance (in.)	**Date**	**5-PA-1**	**5-PA-2**	**5-PA-3**	**5-1N-1**	**5-1N-2**	**5-1N-3**	**Day**
0.0096	1-Mar	0.3295	0.2919	0.2930	0.3282	0.3282	0.3161	0
0.0093	2-Mar	0.3364	0.2984	0.2998	0.3347	0.3346	0.3225	0
0.0097	5-Mar	0.3533	0.3165	0.3172	0.3418	0.3411	0.3291	3
0.0095	9-Mar	0.3837	0.3480	0.3480	0.3620	0.3618	0.3508	7
0.0087	12-Mar	0.3942	0.3578	0.3578	0.3703	0.3703	0.3594	11
0.0084	16-Mar	0.4040	0.3676	0.3674	0.3810	0.3810	0.3699	14
0.0088	23-Mar	0.4190	0.3834	0.3826	0.4019	0.4027	0.3902	21
0.0098	30-Mar	0.4344	0.3994	0.3977	0.4257	0.4281	0.4124	28

Table J.3: Standard and Modified ASTM C 1567 Results for the 10% RHA Samples

Test 1								
Referance (in.)	**Date**	**10-PA-1**	**10-PA-2**	**10-PA-3**	**10-1N-1**	**10-1N-2**	**10-1N-3**	**Day**
0.0101	30-Jan	0.2671	0.3020	0.2846	0.2900	0.2676	0.2788	0
0.0100	31-Jan	0.2746	0.3096	0.2921	0.2975	0.2751	0.2863	0
0.0101	3-Feb	0.3841	0.4143	0.3992	0.3311	0.3075	0.3193	3
0.0102	7-Feb	0.3893	0.4202	0.4048	0.3487	0.3255	0.3371	7
0.0093	11-Feb	0.3907	0.4216	0.4062	0.3563	0.3333	0.3448	11
0.0098	14-Feb	0.3922	0.4233	0.4078	0.3626	0.3396	0.3511	14
0.0087	21-Feb	0.3934	0.4248	0.4091	0.3763	0.3534	0.3649	21
0.0096	28-Feb	0.3973	0.4287	0.4130	0.3971	0.3743	0.3857	28
Test 2								
Referance (in.)	**Date**	**10-PA-1**	**10-PA-2**	**10-PA-3**	**10-1N-1**	**10-1N-2**	**10-1N-3**	**Day**
0.0096	1-Mar	0.3249	0.3256	0.3252	0.2750	0.3075	0.2913	0
0.0093	2-Mar	0.3317	0.3326	0.3322	0.2816	0.3138	0.2977	0
0.0097	5-Mar	0.4477	0.4434	0.4456	0.3131	0.3452	0.3292	3
0.0095	9-Mar	0.4532	0.4491	0.4512	0.3413	0.3721	0.3567	7
0.0087	12-Mar	0.4542	0.4504	0.4523	0.3503	0.3809	0.3656	11
0.0084	16-Mar	0.456	0.453	0.4545	0.3593	0.39	0.3747	14
0.0088	23-Mar	0.4624	0.46	0.4612	0.378	0.4082	0.3931	21
0.0098	30-Mar	0.4709	0.4686	0.4698	0.4021	0.4312	0.4167	28

Table J.4: Standard and Modified ASTM C 1567 Results for the 15% RHA Samples

Test 1								
Referance (in.)	**Date**	**15-PA-1**	**15-PA-2**	**15-PA-3**	**15-1N-1**	**15-1N-2**	**15-1N-3**	**Day**
0.0101	30-Jan	0.3182	0.2972	0.2670	0.2948	0.3348	0.2763	0
0.0100	31-Jan	0.3263	0.3052	0.2749	0.3029	0.3432	0.2836	0
0.0101	3-Feb	0.4899	0.4703	0.4202	0.3474	0.3886	0.3274	3
0.0102	7-Feb	0.5430	0.5295	0.4693	0.3795	0.4221	0.3566	7
0.0093	11-Feb	0.5444	0.5309	0.4706	0.3858	0.4325	0.3640	11
0.0098	14-Feb	0.5456	0.5322	0.4717	0.3934	0.4393	0.3704	14
0.0087	21-Feb	0.5460	0.5330	0.4722	0.4203	0.4483	0.3864	21
0.0096	28-Feb	0.5482	0.5353	0.4742	0.4151	0.4624	0.3903	28
Test 2								
Referance (in.)	**Date**	**15-PA-1**	**15-PA-2**	**15-PA-3**	**15-1N-1**	**15-1N-2**	**15-1N-3**	**Day**
0.0096	1-Mar	0.3243	0.2962	0.3103	0.3165	0.2940	0.3053	0
0.0093	2-Mar	0.331	0.3033	0.3172	0.3231	0.3007	0.3119	0
0.0097	5-Mar	0.5135	0.4921	0.5028	0.3571	0.333	0.3451	3
0.0095	9-Mar	0.5559	0.5327	0.5443	0.4025	0.3753	0.3889	7
0.0087	12-Mar	0.5555	0.5323	0.5439	0.4132	0.3853	0.3992	11
0.0084	16-Mar	0.5566	0.5334	0.5450	0.4212	0.3928	0.4070	14
0.0088	23-Mar	0.5587	0.5354	0.5471	0.4325	0.4033	0.4179	21
0.0098	30-Mar	0.5624	0.5390	0.5507	0.4464	0.4163	0.4314	28

Table J.5: Standard and Modified ASTM C 1567 Results for the 20% RHA Samples

Test 1								
Referance (in.)	**Date**	**20-PA-1**	**20-PA-2**	**20-PA-3**	**20-1N-1**	**20-1N-2**	**20-1N-3**	**Day**
0.0101	30-Jan	0.2794	0.2808	0.2574	0.2743	0.2868	0.2476	0
0.0100	31-Jan	0.2886	0.2897	0.2663	0.2832	0.2956	0.2559	0
0.0101	3-Feb	0.3013	0.3003	0.2768	0.2991	0.3073	0.2684	3
0.0102	7-Feb	Samples Broken			0.3320	0.3324	0.2941	7
0.0093	11-Feb	X	X	X	0.3570	0.3509	0.3133	11
0.0098	14-Feb	X	X	X	0.3743	0.3646	0.3270	14
0.0087	21-Feb	X	X	X	0.4038	0.3990	0.3553	21
0.0096	28-Feb	X	X	X	0.4337	0.4280	0.3814	28
Test 2								
Referance (in.)	**Date**	**20-PA-1**	**20-PA-2**	**20-PA-3**	**20-1N-1**	**20-1N-2**	**20-1N-3**	**Day**
0.0096	1-Mar	Samples Broken						0
0.0093	2-Mar	X	X	X	X	X	X	0
0.0097	5-Mar	X	X	X	X	X	X	3
0.0095	9-Mar	X	X	X	X	X	X	7
0.0087	12-Mar	X	X	X	X	X	X	11
0.0084	16-Mar	X	X	X	X	X	X	14
0.0088	23-Mar	X	X	X	X	X	X	21
0.0098	30-Mar	X	X	X	X	X	X	28

K. SEM AND EDX IMAGES OF RHA 1567 MORTAR BAR SAMPLES

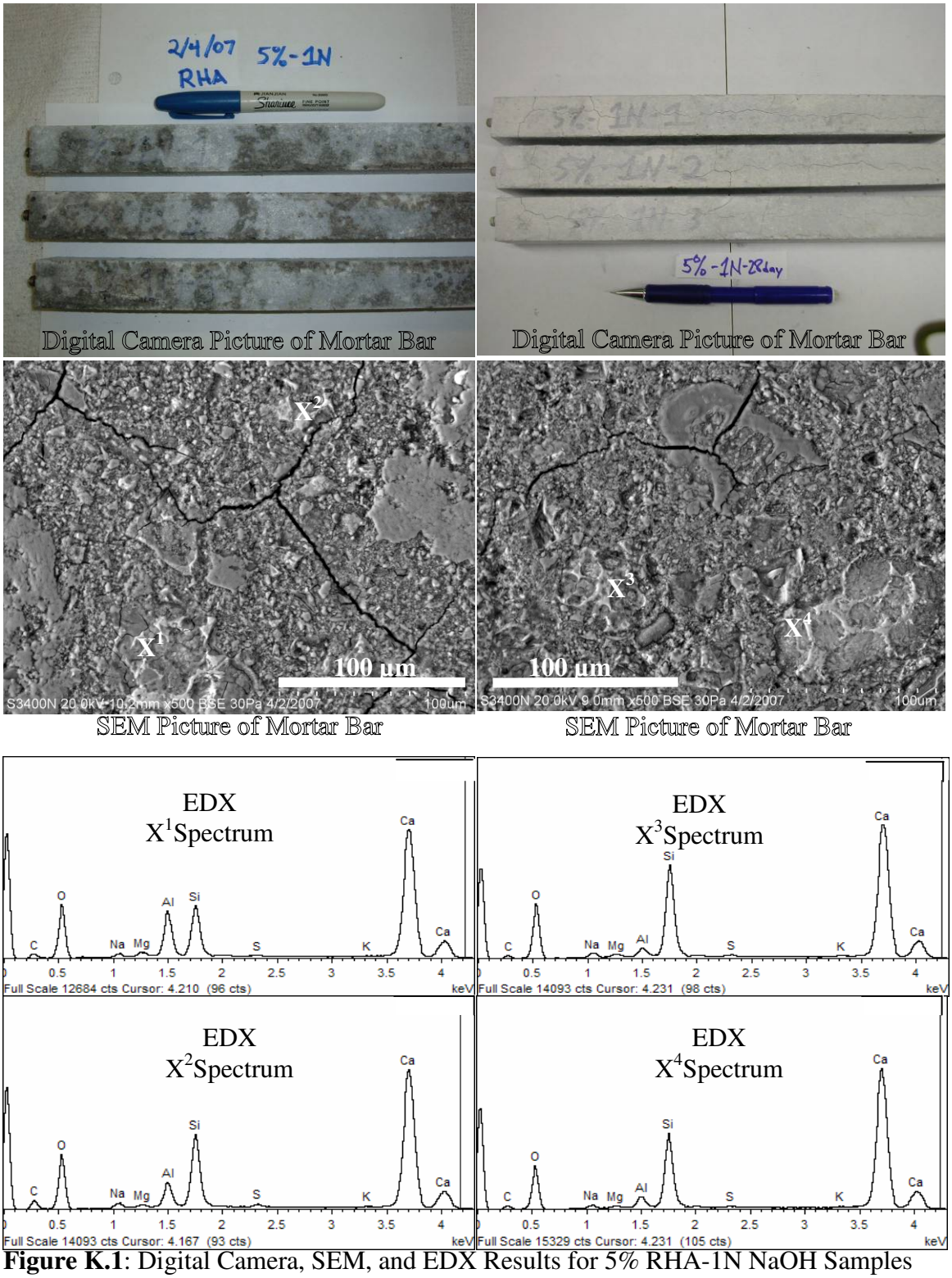

Figure K.1: Digital Camera, SEM, and EDX Results for 5% RHA-1N NaOH Samples

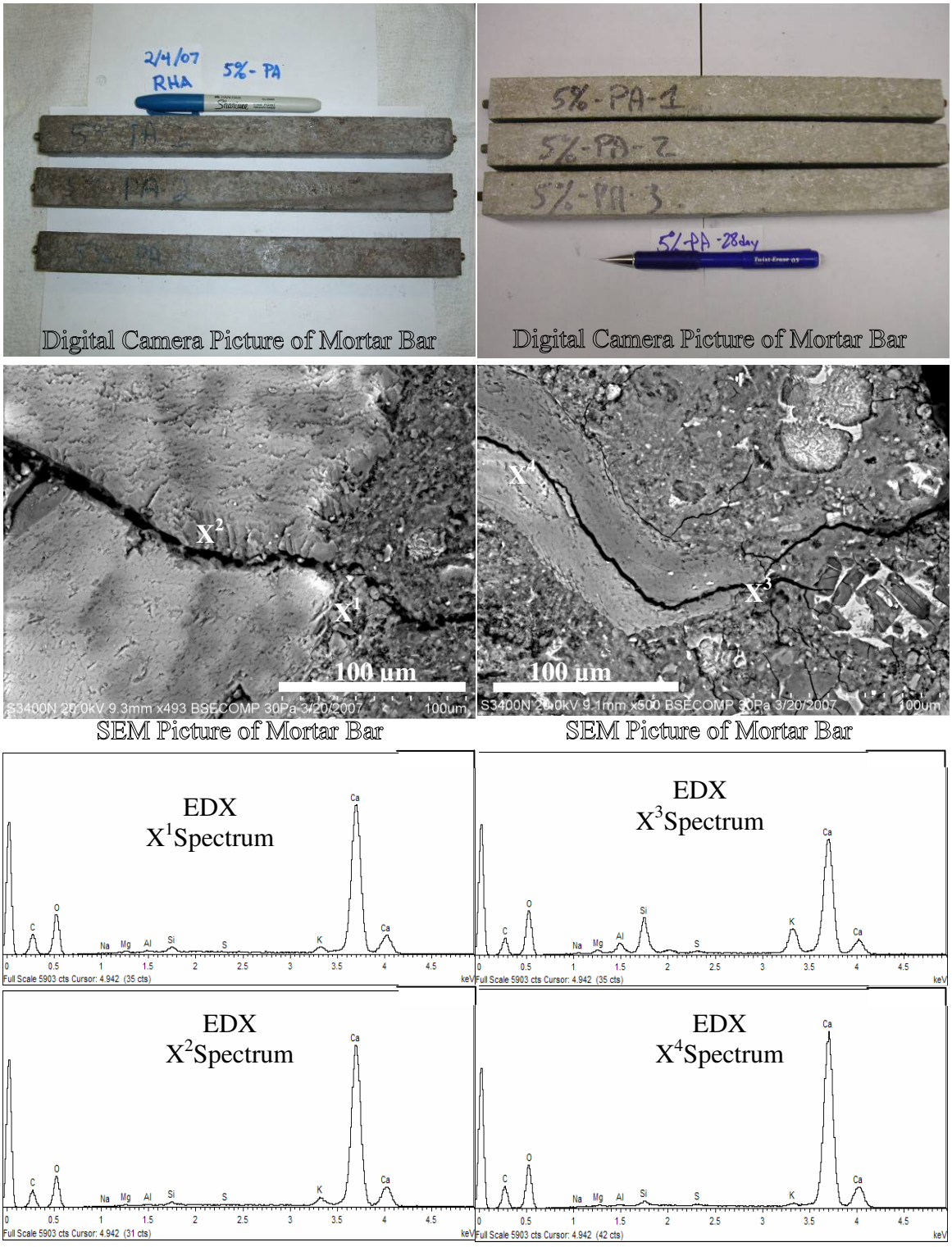

Figure K.2: Digital Camera, SEM, and EDX Results for 5% RHA-Potassium Acetate Samples

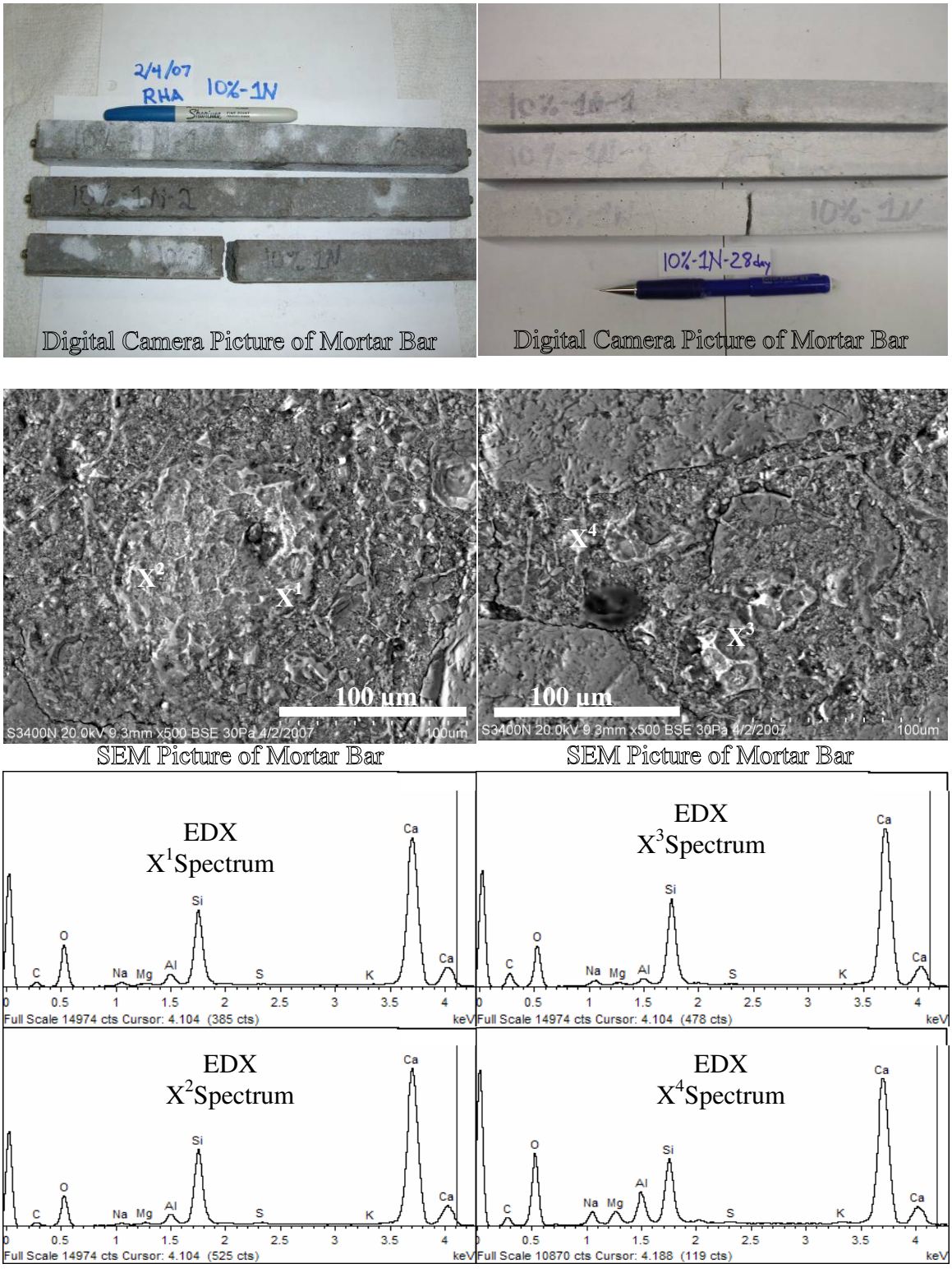

Figure K.3: Digital Camera, SEM, and EDX Results for 10% RHA-1N NaOH Samples

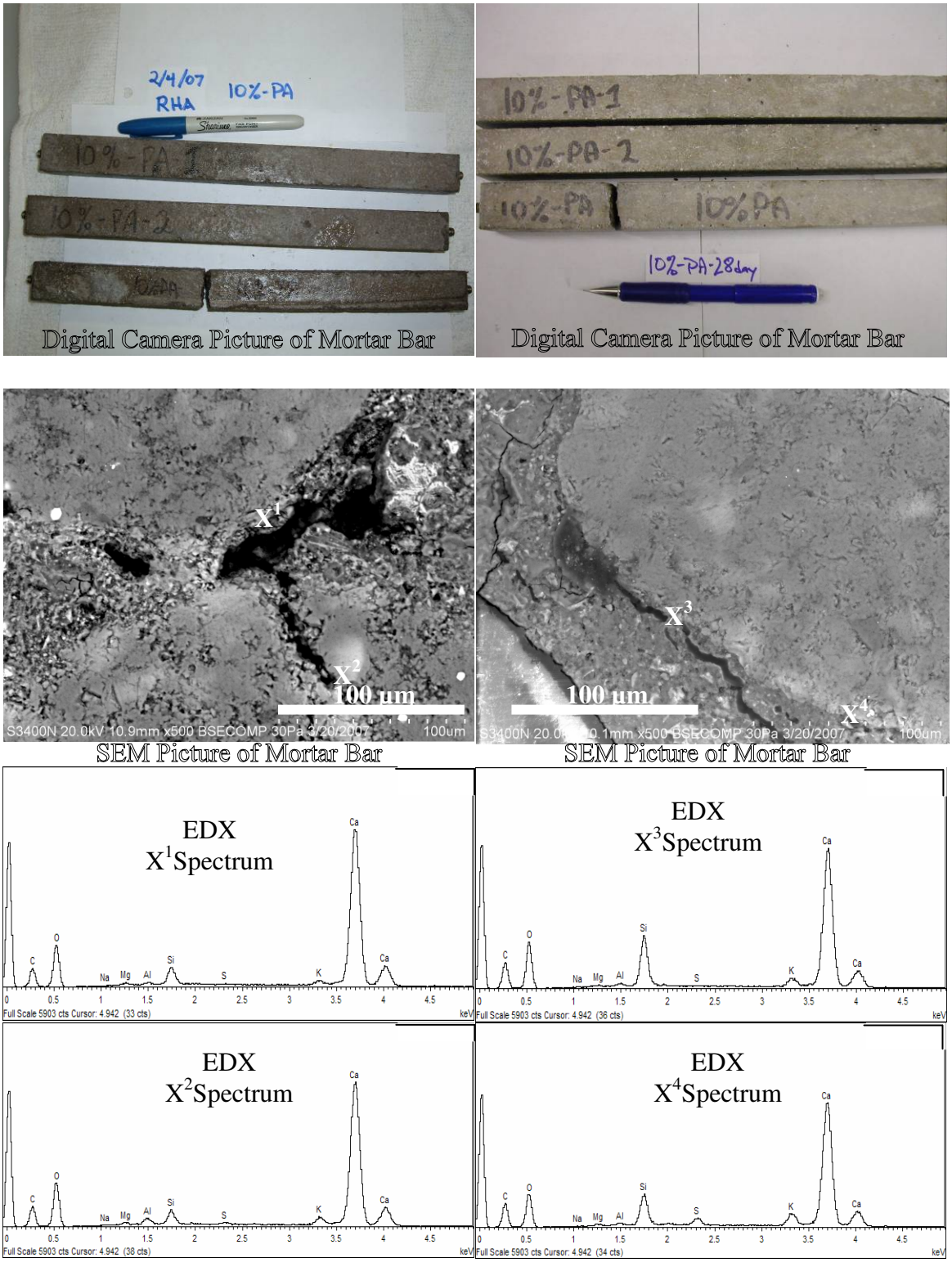

Figure K.4: Digital Camera, SEM, and EDX Results for 10% RHA-Potassium Acetate Samples

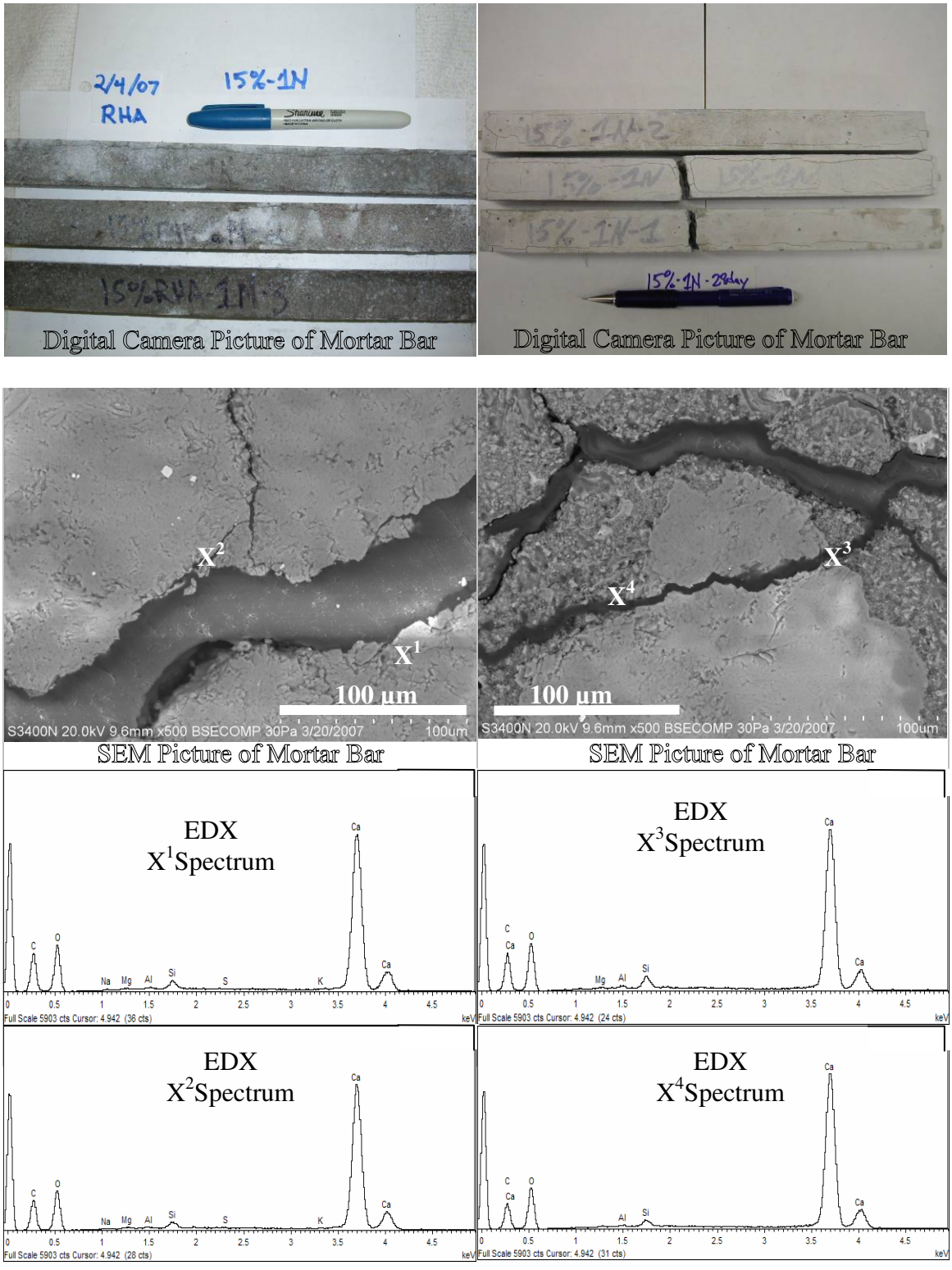

Figure K.5: Digital Camera, SEM, and EDX Results for 15% RHA-1N NaOH Samples

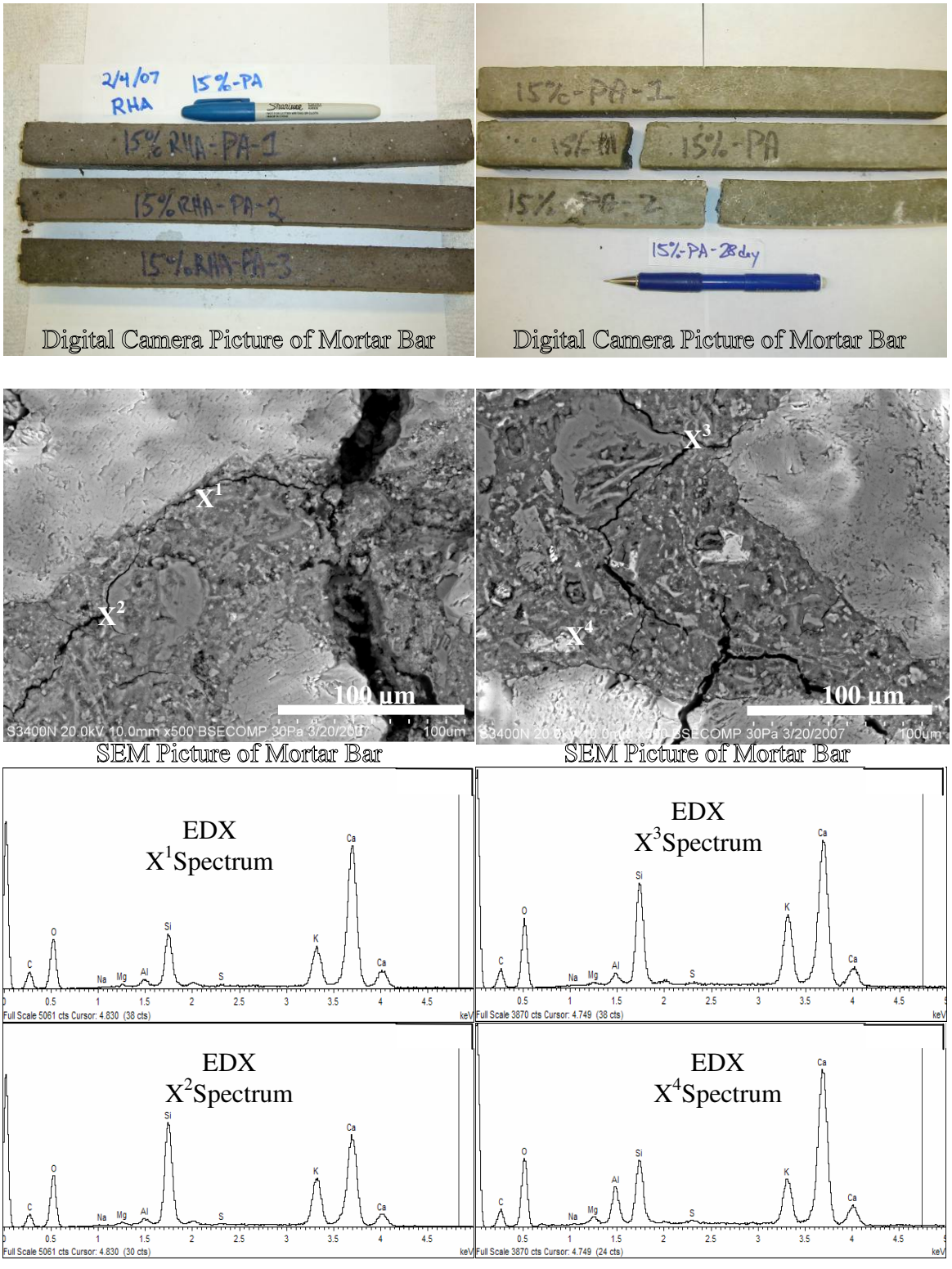

Figure K.6: Digital Camera, SEM, and EDX Results for 15% RHA-Potassium Acetate Samples

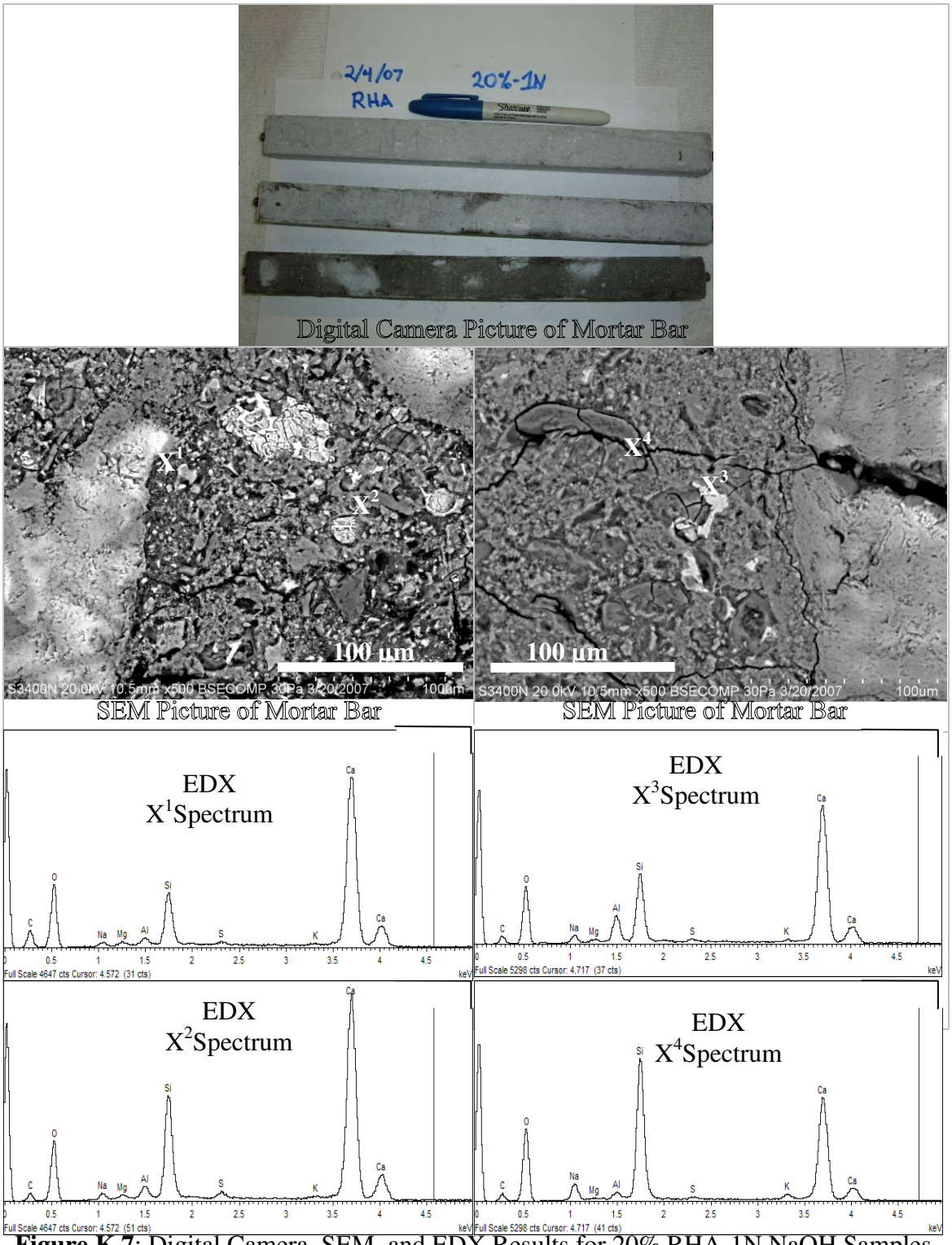

Figure K.7: Digital Camera, SEM, and EDX Results for 20% RHA-1N NaOH Samples

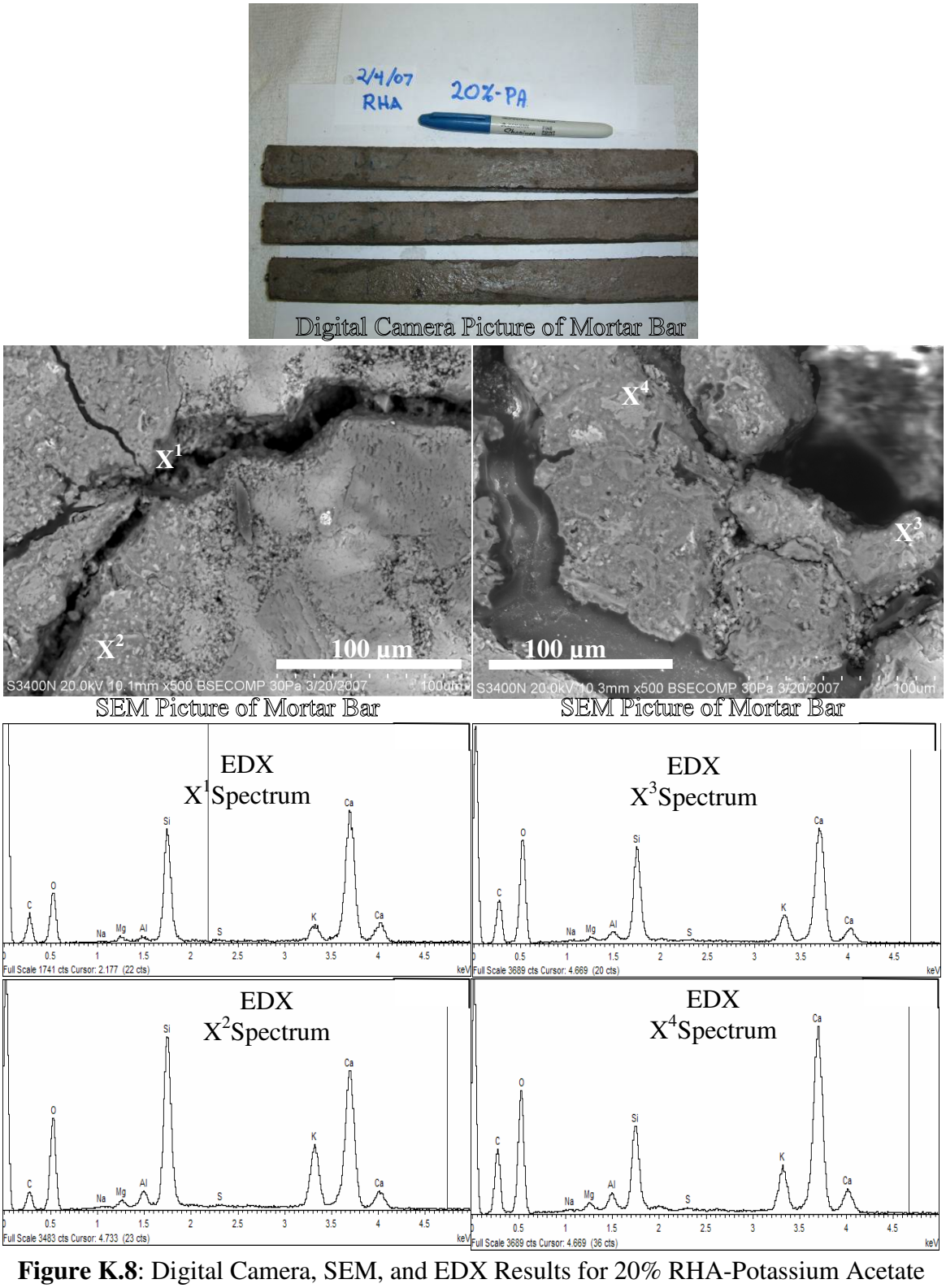

Figure K.8: Digital Camera, SEM, and EDX Results for 20% RHA-Potassium Acetate Samples